FINAL REPORT

Medium Caliber Lead-Free Electric Primer

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14. ABSTRACT
Lead Free Electric Primers (LFEP) based on energetic nanocomposites were examined as a replacement for conventional lead styphnate primers. On small scale samples, outstanding all-up round action times (AUR-ATs) were obtained for ambient (2.92 ms) and at low-temperature (3.02 ms). Large scale batch verification testing failed to meet the AUR-AT requirement of less than 3.5 ms as specified in MIL-DTL-1394G. The AUR-ATs were affected by the very long fabrication time for the primers. Usually, primer materials were made, primers loaded and rounds built in a period of 1 week. Due to technical issues, the final 500 test rounds were assembled over a period of 7 months.

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List of Acronyms and Symbols

ADN	Ammonium Dinitramide
Al	Aluminum
Alex®	Trademark name for Argonide's Exploded Aluminum
AN	Ammonium Nitrate, NH_4NO_3
AP	Ammonium Perchlorate, NH_4ClO_4
ARDEC	Armament Research, Development and Engineering Center
AUR	All-Up Round
AUR-AT	All-Up Round Action Time/s
BET	Brunauer-Emmett-Teller
BM	Ball Mill
BPR	Ball to Product Ratio
BTATZ	3,6-bis(1H-1,2,3,4-tetrazol-5-amino)-s-tetrazine
C	Capacitance
CAD	Cartridge Actuated Device
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CP	Chevron-Phillips
DABT	Diammonium Bitetrazole
DBX-1	Copper 5-Nitrotetrazole
DDNP	Diazodinitrophenol
DMF	Dimethyl Formamide
DOD	Department of Defense
DOE	Department of Energy
DTRA	Defense Threat Reduction Agency
E	Energy
ECP	Engineering Change Proposal
EHS	Environmental Health and Safety
EMPI	Energetic Materials & Products Inc.
ENC	Energetic Nanocomposite
EPA	Environmental Protection Agency
ESD	Electro Static Discharge
$E_{50\%}$	Energy required for at least 50% positive ignition
E_{onset}	Minimum onset energy needed to initiate material combustion or deflagration
E_{sp}	Spark Energy
ESOH	Environment, Safety, and Occupational Health
ESTCP	Environmental Strategic Technology Certification Program
FC	Fluorocarbon
GD-OTS	General Dynamics Ordnance and Tactical Systems
HERO	Hazards of Electromagnetic Radiation to Ordnance
i-PrOH	Isopropyl Alcohol or Isopropanol
KDNP	4, 6-dintro-7-hydroxybenzofuroxan
Kel-F®	Trademark name of DuPont for Polychloro trifluoroethylene
KOH	Potassium Hydroxide
LANL	Los Alamos National Laboratory
LFEP	Lead-Free Electric Primer

LCAAP	Lake City Army Ammunition Plant
LLNL	Lawrence Livermore National Laboratory
LT	Low Temperature
MIC	Metastable Interstitial Composite
MIL-DTL	Military Standard
MoO_3	Molybdenum Trioxide
NAAQS	National Ambient Air Quality Standard
NaOH	Sodium Hydroxide
NASA	National Aeronautics and Space Administration
NAVSEA	Naval Sea System Command
NAWCWD	Naval Air Warfare Center Weapons Division
Neoflon®	Tradename of Daikin Industries of Japan for Polychloro trifluoroethylene
NESDI	Navy Environmental Sustainability Development to Integration
NJIT	New Jersey Institute of Technology
NSWC-IH	Naval Surface Weapon Center – Indian Head
OSHA	Occupational Safety and Health Administration
OPNAVINST	Office of the Chief of Naval Operations Instruction
PCTFE	Polychlorotrifluoroethylene
PE	Polyethylene
PEO-W	Program Executive Officer – Weapons
PMA	Program Management Activity
P_{max}	Peak Maximum Pressure
PVAT	Pressure-Velocity All-Up Round Action Time
PQL	Product Quality Laboratory
RAM	Resonance Acoustic Mixer
RCRA	Resource Conservation and Recovery Act
SDWA	Safe Drinking Water Act
SEM	Scanning Electron Microscopy
SERDP	Strategic Environmental Research & Development Program
SMCA	Single Manager for Conventional Ammunition
SOP	Standard Operating Procedure
TSCA	Toxic Substance Control Act
TGA	Thermogravimetric Analysis
TNR	Trinitroresorcinol
V	Voltage
VDC	Voltage Direct Current
WSESRB	Weapons System Explosive Safety Review Board

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EXECUTIVE SUMMARY

The objective of this project was to validate that a Lead-Free Electric Primer (LFEP) based on a nano energetic composite could be used to replace the conventional M52A3B1 primer for medium caliber ammunition while meeting MIL-DTL-1394G requirements. The primary performance objective was meeting the all-up round action time (AUR-AT) requirement of less than 4.0 ms at ambient and low-temperature (-65°F). The program was supposed to optimize a small scale LFEP composition based on AUR-ATs, develop a scalable process for LFEP synthesis, demonstrate the scaled-up LFEP met DOD AUR-ATs, automate the LFEP loading and pressing steps, demonstrate LFEP reliability in a Gatling Gun firing of 505 rounds, and transition the technology to the DOD and industry.

At the beginning of this ESTCP project, a number of obstacles arose including the need to re-formulate the LFEP composition. Three of the 5 components used in the previous SERDP project, Al(50nm), BTATZ, and Kel-F, had to be replaced with commercially available material. The ability to develop and characterize new Energetic Nano Composites (ENCs) was mainly performed at Universities and DOE National Laboratories where ENCs were tested using pressure cells and burn tubes. Therefore changing the ENC composition, finding a new gas additive and suitable binder for the LFEP was a daunting task.

To address this need, NAWCWD developed a new laboratory tool that enabled the rapid optimization of ENCs and LFEP compositions, called the "Al Pan Dent Test". This test helped identify a wide range of other useful ENCs that could be used to make LFEP composites, igniters, and pyrotechnics. The Al/MoO_3 ENC turned out to be one of the poorer performing composites in the Al Pan Dent Test. However, a LFEP composition using this ENC was developed, 66% Al(80nm)/MoO_3, 30% AN, 2% Kel-F, 2% C, that met DOD requirements. The synthesis process was simplified to eliminate most hazardous steps but the dry addition of carbon to the Al/MoO_3/AN/Kel-F was essential. The carbon is only needed for electric ignition systems but for percussion systems the Al/MoO_3/AN/Kel-F is suitable candidate. This standard composition when prepared and consolidated correctly gave typical AUR-ATs of 3.05 $\pm$ 0.08 ms at ambient and 3.12 $\pm$ 0.08 ms at -65 °F. Good AUR-ATs were obtained for compositions with AN in the weight percent range of 25 to 38%. The Al(80nm)/MoO_3(45nm) molar ratio of 2.63 to 2.69 was found to produce the lowest AUR-ATs at both ambient and low-temperatures.

To ensure starting material quality and LFEP reliability, a list of recommended characterizations was developed for each of the starting materials and can be found in Table 6.2.

A significant advancement was made in the area of nanoenergetic composites. Prior to this program, ENCs were generally made on a 1-2 g scale by sonication. A scalable process was developed to prepare ENCs and the scaled-up materials appear to be superior to the small scale sonicated materials. Many 100g batches have been prepared and characterized. Over 2 Kg of ENC have been prepared and tested using the scale-up method developed in this program for DTRA applications. The labor cost of preparing ENCs has been drastically reduced but starting material costs are still an issue. Multi-kilogram batches could be easily made in an industrial setting.

A scaled-up 100g batch of the LFEP composite was successfully prepared in the laboratory and had an average AUR-AT of 3.21 ms. However, the final step of carbon addition to the Al/MoO$_3$/AN/Kel-F powder failed in our scale-up facility. Very poor mixing led to a very heterogeneous composite due to a design flaw in our mixing apparatus. The flaw can be easily fixed and the redesign was tested in the laboratory but has yet to be demonstrated on the 100g scale. The final dry addition of carbon is achievable in an industrial environment on a multi-kilogram scale. **The LFEP was successfully scaled-up in our laboratory but not at our scale-up facility.**

A schematic for the LFEP preparation is shown below:

$$Al(80nm) + MoO_3 \xrightarrow[\text{hexane}]{\text{Ball Mill}} Al/MoO_3 \xrightarrow{+AN} Al/MoO_3/AN \xrightarrow{+Kel\text{-}F/hexane} \xrightarrow{-hexane} Al/MoO_3/AN/Kel\text{-}F \xrightarrow{+C} Al/MoO_3/AN/Kel\text{-}F/C$$

A powder loading system in combination with the 5x5 multi-die set have been demonstrated using the LFEP. The powder loading system was used to fill one primer cup at a time but an automated system could be developed to reduce labor costs. The multi-die set configuration may also reduce labor cost by combining the cutting of the seal paper and pressing in one step and by pressing 25 primers at once. Although the powder loading system and multi-die set was demonstrated with LFEP material, the AUR-ATs averaged almost 4 ms. Additional testing is needed to identify critical parameters and lower the AUR-ATs to acceptable limits. **The powder loading system and multi-die pressing system have been tested but not validated.**

A working LFEP material has been developed, validated on a small scale, scaled-up and a viable primer loading system tested that could be transitioned to the industry. Due to funding issue and capability realignment issues, the final testing was not performed under ESTCP funding. Funding was secured from the Navy Environmental Sustainability Development to Integration (NESDI) Program for primer loading and pressing and support was obtained from PMA-242 to pay for the firing and data collection. A 200g batch of LFEP was prepared, 40 20 mm rounds built-up and Mann Barrel gun firing produced acceptable AUR-ATs averaging 3.21 ms. A total of 505 primers were loaded into primer cups in Sept. 2012 (after 4 month of storage in hexane), 505 20 mm rounds built at Picatinny Arsenal in May 2012 (after 5+ months of primer cup storage at Picatinny Arsenal) and fired in Sept. 2012. The rounds failed to meet the 3.5 ms AUR-AT. The data set has not been received at this point but is expected by Nov. 2012 from PMA-242. Normally, the preparation of the LFEP material is followed by the primer loading & pressing, and then the 20 mm rounds are built-up. This entire process usually is performed within 5 working days. Due to lack of funding and loss of capabilities issue due to Base Re-alignment and Closure the process took close to 10 months. The primer material was made and stored in hexane in a polyethylene bottle for 4 months before the 505 primers were made. They were shipped off to Picatinny Arsenal where the primers sat for over 5 months before round build-up. The LFEP aged during the storage in hexane or while the unprotected primers awaited build-up at Picatinny Arsenal. The PMA-242 transition of this technology will not occur due to final testing failure.

1.0 INTRODUCTION

Primers and detonators are widely used throughout the Department of Defense (DOD) to initiate different types of ordnance. Primers and detonators are part of an explosive train where the primers/detonators initiate a secondary explosive or propellant. In most cases, the primers or detonators are based on primary explosives, either lead styphnate or lead azide. These lead containing primers are sources of airborne, water, and soil contamination.

1.1   BACKGROUND

The DOD currently fires in excess of 3 billion rounds of small and medium caliber ammunition per year in training and in operations. The ignition systems for these rounds are lead-based (lead styphnate) primary explosives that are initiated by impact (percussion) or by spark (electric). The primer reacts to produce heat and hot products that ignite the propellant bed. The burning propellant produces high temperatures and pressures that ultimately launch the projectile. The DOD's primers produce about 165 tons of air-borne lead that ultimately contaminates ranges, ground water, flora and fauna. Lead is a toxic heavy metal that damages the central nervous, cardiovascular and immune systems. Children are highly sensitive to lead and lead exposure has been cited for reduced intelligence.[1] Lead compounds have been classified as a group 2A carcinogen by the International Agency for Research on Cancer. Lead is the environment is persistent because airborne lead is deposited and strongly absorbed in soil. The low solubility of lead generally traps lead in the upper soil layers. However, lead does become mobile on acidic soils and with acid rain where the lead can leach into ground water.

In order to reduce airborne lead emission from DOD ammunition, a drop-in lead-free electric primer (LFEP) replacement (material substitution) for the Mix FA-874 used in the M52A3B1 primer was investigated. The M52A3B1 primer is used in all electrically initiated 20 mm rounds. The M52A3B1 primer is used in the M50 and M60 series of 20 mm Gatling guns on the Navy's F/A-18 and Air Force's F-22 aircraft and in the M197 Gatling gun used on Army and Navy helicopters. It was also used in the MK-15 Phalanx Close-In Weapon System for ship protection.

In the early 1990's, Los Alamos National Laboratory (LANL) developed a new class of nano layered energetic material called "Metastable Interstitial Composite" (MIC) prepared by ultra-high vacuum magnetron sputtering.[2] Alternating nano layers (< 100 nm) of aluminum and copper oxide were prepared and characterized.[3] The thin nanolayers increased the surface area contact between the fuel (aluminum) and oxidizer (copper oxide) by close to 1000x as compared to conventional micron thermite powders resulting in significantly higher reaction rates. By 1998, LANL and the Naval Air Warfare Center Weapons Division (NAWCWD) patented the idea of percussion primers based on MIC materials.[4] In the early 2000's, LANL developed a method to prepare nano aluminum powders by aluminum vapor condensation. The nano aluminum powders were then used to produce energetic nanocomposites (ENCs) by ultra-sonication methods.[5] ENCs were also prepared by mechanical milling processes such as "Arrested Reactive Milling"[6] and by a sol-gel process developed at Lawrence Livermore National Laboratory (LLNL).[7] The fast reaction rates (< 100 μs), low temperature reactivity (-78°C), high impact and electrostatic discharge sensitivities, high energy content and high thermal stabilities (> 300°C) properties make them ideal for primer applications. These ENCs have been

called MIC, Super-Thermite, Nano-Thermite and Nano-Structured Energetic Materials. An example of the reaction of an ENC is between aluminum and molybdenum trioxide (MoO_3) shown in equation 1:

$$2\ Al + MoO_3 \rightarrow Al_2O_3 + Mo \qquad\qquad \Delta H = -4.7\ kJ/g\ (-18.3\ kJ/cc) \qquad\qquad (1)$$

The nanocomposite reacts between 100 to 1000 times faster than traditional micron thermite composites and its energy content is superior to lead styphnate (-1.83 kJ/g and -4.49 kJ/cc). One of the main deficiencies of nanocomposites is the low quantity of gaseous products, thus low potential to perform work. Gases are needed to push the hot ENC reaction products onto the propellant bed for fast propellant ignition.

The US Army and Navy successfully demonstrated primers based on ENCs for small caliber ammunition and igniters for Cartridge Actuated Devices and Propellant Actuated Devices by the US Army and US Navy.[8,9] Lead-Free primers were also investigated for use in percussion initiated medium caliber ammunition by the Army[10] and electrically initiated medium caliber ammunition by the Navy.[11]

NAWCWD's Lead-Free Electric Primer effort began as a Strategic Environmental Research and Development Program (SERDP) Exploratory Development (SEED).[12] The objective of the SEED program was to demonstrate the feasibility of MIC based Lead-Free Electric Primers. The SEED program initially started with the concept of substituting an electrically conductive MIC material for lead styphnate that is currently used in the M52A3B1 medium caliber electric primer. Investigators discovered early in the SEED effort that the direct substitution of a MIC-based primer mix for the normal lead based formulation did not produce reliable "ohmic heating" ignition. There were a number of unexplained phenomena and unresolved issues brought to light during this early feasibility effort. It was observed that the electrical conductivity of the MIC material used in the primer mix significantly changed over a relatively short interval of time. The initiation of MIC-based primers was highly variable, working one day and not the next. Resolution of these and other fundamental questions was critical to the success of the follow-on effort.

The full SERDP program entitled, Lead-Free Electric Primer, began in 2002, which included the U.S. Army as well as other U.S. Navy and Department of Energy (DOE) laboratories. The Navy focused their attention on establishing a lead-free primer design as a substitute for the current 20-mm M52A3B1 primer. The Army investigated the use of MIC-based primer material in the low impedance PA520 electric primer used in the lightweight 30-mm gun system employed on the AH-64 Apache helicopter. The DOE and Navy laboratories leveraged the expertise and capabilities of organizations involved in ENC research. They served as a valuable resource of information on ENC technology. The overall LFEP program was structured around three twelve-month long phases with relatively simple objectives. Phase I focused on acquiring the necessary materials to produce several different ENC compositions and evaluated their basic properties through laboratory testing and limited test firing operations. Phase II was designed to thoroughly evaluate the candidate primer compositions under laboratory conditions and develop safe mixing and pressing procedures, as well as perform a limited number of all-up-round (AUR) firing tests. The final Phase III effort was designed to continue the evaluation process and through

statistically significant test processes, arrive at a single primer design that could serve as a suitable entry point into a subsequent design and development program.

The Lead-Free Electric Primer program had many successes:
1) Optimized LFEP formulation composed of 71% Al(50nm)/MoO$_3$, 25% 3,6-bis(1H-1,2,3,4-tetrazol-5-amino)-s-tetrazine (BTATZ), 2% Kel-F and 2% carbon.
2) Demonstrated All-Up Round Action Times (AUR-ATs) as low as 2.87 ms.
3) Developed a laboratory method, "The Al Pan Dent Test", to test the ENC powders (Al(50nm)/MoO$_3$ and Al(50nm)/Bi$_2$O$_3$) and correlated the Deflection number to LANL's Standard Peak Pressure measurements.
4) Characterized starting materials by a variety of analytical tools.
5) Determined the source of MoO$_3$ aging and photosensitivity properties.
6) Developed a heat treatment process to eliminate the MoO$_3$ aging and photosensitivity problems.
7) Identified Ammonium Dinitramide (ADN) as a suitable alternative gas additive.
8) Developed an Electrostatic Discharge (ESD) tester that could measure down to less than 1 microjoule.
9) Optimized LFEP preparation process to reduce hazardous steps while maintaining high primer performance.
10) Developed a suitable backup LFEP formulation based on Al/AgIO$_3$, BTATZ, Kel-F and carbon.
11) Demonstrated that LFEP could be made using Al(50nm)/MoO$_3$ from different sources including Nanotechnology, Naval Surface Warfare Center, Indian Head (NSWC-IH), LANL, and NAWCWD.
12) Started initial evaluation of replacing Al(50nm) powders with Al(80nm).

The LFEP SERDP project ended with a number of issues and basically a brand new LFEP formulation was needed:
1) BTATZ was not a viable gas additive since it was not commercially available. A replacement was needed.
2) Kel-F, a Polychorotrifluoro ethylene (PCTFE) polymer, which was originally made by DuPont was being made only in Japan. A replacement binder was needed.
3) The vendor of 50 nm Al powders, Nanotechnology, went out of business so commercial 50 nm Al was no longer available. The 80 nm Al from Technanogy was a replacement option but an optimized LFEP formulation with low AUR-ATs needed to be demonstrated. In the late stages of the SERDP program, the LFEP formulation KTHW24 using 80 nm Al powders gave unacceptable AUR-ATs > 4.4 ms at low temperatures. In comparison, the same formulation using 50 nm Al, KTHU22, gave low temperature AUR-ATs of < 3.21 ms.

There was no method to scale-up the ENC powder to 100 g quantities. The ultra-sonication method used enabled the preparation of only 2 g. NSWC-IH had a sonication process for 10 g batches and Lawrence Livermore had a sol-gel process to prepare 2 g quantities. All known methods were expensive and labor intensive.

In the Environmental Strategic Technology Certification Program (ESTCP) 20 mm rounds were prepared using the LFEP, and evaluated by capturing live fire data in an outdoor range. Barrel pressure, projectile velocity, and Action Times (PVAT) were collected on the instrumented

Mann Barrel gun. The material substitution of the FA-874 mix with a LFEP will eliminate the lead emissions from the DOD's 20 mm electrically initiated ammunition and reduce lead exposure to military personnel, ammunition workers, range personnel, and the civilian population. The material replacement of the lead containing FA-874 primer mix will benefit the DOD by reducing lead contamination of in-door and out-door ranges where some have been shut down for expensive clean-up operations and cause training delays.

1.2   OBJECTIVE OF THE DEMONSTRATION

The objective of this effort was to demonstrate that environmentally friendly primers based on energetic nanocomposites could replace the lead (Pb) based M52A3B1 primers used in 20 mm ammunition while meeting DOD's all-up round action-time requirements at ambient and low-temperatures (-65 °F) as described in MIL-DTL-1394G. A critical issue was to demonstrate that the new LFEP formulation could be scaled-up for production and that the scale-up process would not detrimentally impact the primer performance. At the onset of the ESTCP program, only small scale preparation methods were known for energetic nanomaterials. In the previous SERDP program on LFEP, primer materials were prepared and tested in only 1 to 5 gram batch sizes. A scalable technology was needed to enable industrial production at kilogram levels. Another critical issue was to introduce new technologies to improve the loading and consolidation processes.

The overarching objective was to demonstrate that LFEPs could be made in quantity, to develop processes to enable commercial production, to demonstrate live-fire in a single fire Mann Barrel and in a rapid fire Gatling gun, and to transition developed technologies to Program Management Activity (PMA) 242, the Army Medium Caliber Ammunition, and commercial production. The primary benefit to the Navy is for Gatling guns in the F/A-18 and Navy helicopters, for the Air Force it is for their F-22 Fighter, and Army for their helicopters. The goal of the program is to eliminate the lead styphnate and barium nitrate components in the current 20mm electric primer for the purposes of protecting our troops, production workers and our environment.

Mix FA-874 used in the M52A3B1 primer is an environmental, safety, and occupational health risk. Production of the current M52A3B1 primer creates solid and liquid hazardous waste streams and exposes factory workers to lead based compounds. The production of DOD ammunition occurs at Lake City Army Ammunition Plant (LCAAP) in Missouri. LCAAP makes the Mix FA-874 in a series of reaction and addition steps. First trinitroresorcinol (TNR) is made by nitrating their resorcinol feedstock. Magnesium oxide is added to TNR to produce Magnesium Bis(trinitroresorcinol).   Lead   nitrate   is   added   to   the   magnesium bis(trinitroresorcinol) to form lead styphnate and magnesium nitrate. The additional ingredient, barium nitrate, calcium silicide, TNR, Gum Arabic and acetylene black are added to the lead styphnate in water. A 10 pound ball of primer material with about 25 weight percent water is made for the primer loading process. The primers are generally processed into all up rounds within 24 hours. The wet primer mix is hand pressed into a plate with holes to form primer pellets. The pellets are transferred into primer cups and then heated in an oven to remove excess water and then pressed/consolidated.

Lead contaminated waste water streams are created by workers who manually handle the wet

primer mixes where scraps fall onto wet floors and by the chemical production of the primer mix. In addition, workers clothing can be easily contaminated by workers directly handling wet primer mixes. The composition of the Mix FA-874 used in the M52A3B1 primer is listed in Table 1:

Table 1: Composition of Mix FA-874 Used by Lake City in the M52A3B1 Primers

Mix FA-874	Components	Percentage
Used in M52A3B1	Lead Styphnate	37.5-42.5
	Barium Nitrate	41.75-46.75
	Calcium Silicide	10.5-15.5
	Trinitroresorcinol	1.25 max
	Acetylene Black	1 max
	Gum	0.75-1.25

The replacement of the standard lead primer mixes with Lead-Free materials will resolve some Environment, Safety, and Occupational Health (ESOH) risks by eliminating lead contaminated water streams and potential worker exposure to lead by eliminating direct handling of lead primer mixes at the LCAAP. The most common exposure to lead comes from inhalation or ingestion at manufacturing facilities, during de-militarization (open burn, open detonation), and during life fire. Lead leaching from DOD ranges into the ground water may contaminate the drinking water of the surrounding communities. The Environmental Protection Agency (EPA) has set the acceptable lead contamination level in drinking water at 0. Lead contamination of drinking water from DOD ranges is unacceptable. The elimination of lead from DOD primers will benefit DOD indoor and outdoor ranges, PMA 242, Program Executive Officer - Weapons (PEO-W), and reduce life cycle costs by reducing disposal costs. A simple base hydrolysis can be used to deactivate the LFEP material and this process will generate a relative benign waste stream containing alumina, molybdenum trioxide, and a nitrate.

1.3   REGULATOR DRIVERS

The DOD use of lead containing primers results in airborne lead that also contaminates the water and soil. The airborne lead emission is covered by the **Clean Air Act**. In October, 2008 under the Clean Air Act, the EPA re-evaluated the toxicity of lead and reduced the airborne lead limit for the National Ambient Air Quality Standard (NAAQS) from 1.5 $\mu g/m^3$ down to 0.15 $\mu g/m^3$. This 10X reduction in the limit of airborne lead required new equipment to be installed to monitor at the new lower limit. Each 20 mm round using the M52A3B1 primer emits 33.5 mg of airborne lead and a M61A2 Gatling gun at 6000 rounds per minute emits 3.35 g of lead per second. The M197 Gatling gun firing at a rate of 720 rounds per minute emits 0.4 g of lead per second. The Phalanx firing at a rate of 4800 rounds per minute emits 2.68 g of airborne lead per second. These weapons far exceed the NAAQS for airborne lead during life fire operations exposing DOD employees to very high levels of airborne lead. Sources of lead contamination of water include the airborne lead settling onto the ground and contaminating ground water and waste water streams created from the manufacturing process of lead based primers. Lead contamination of water is covered by the Clean Water Act and Safe Drinking Water Act (SDWA) while ground water contamination is covered by provisions in the SDWA, Resource Conservation and Recovery Act (RCRA), and the Comprehensive Environmental Response,

Compensation, and Liability Act (CERCLA). The solid and liquid hazardous waste generated in the production of lead primers and solid hazardous waste from clean-up of indoor and outdoor ranges are covered by RCRA, CERCLA and the DOD Hazardous and Solid Waste Minimization Amendment of 1984.

The manufacturing and firing of lead based ammunition is currently an ESOH risk. DOD personnel are exposed to airborne lead during training, in live fire exercises, and during indoor and outdoor range cleanup. Personnel involved in the cleanup of indoor ranges have been found to have lead blood levels 10 times higher than the allowable limit. Elevated lead blood levels have been observed in personnel who work or frequently visit indoor gun range.[13] Lead is also an environmental issue for the manufacturers of DOD ammunition where personnel are exposed to lead by direct manipulation of lead-based primer materials and they also generated lead contaminated water streams that have to be disposed of as hazardous waste. In 1993, **Executive Order 12856** Federal Compliance with Right-to-Know Laws and Pollution Prevention Requirements instructed federal facilities to reduce procurement of hazardous substances and chemicals, such as lead, and to promote the acquisition of environmentally friendly products. Although the EPA does not have regulatory purview over ammunition, the **Toxic Substance Control Act** (TSCA) can regulate the ingredients if environmentally friendly alternatives are available. The EPA could require the use of new lead-free primer alternatives in the near future. The DOD has enacted regulations that restrict the use of lead. The Operational Navy Instruction (OPNAVINST) 4110.2 on Hazardous Material Control and Management and Energetic Production Pollution Prevention instruction 3.1.6.C regulate the use of lead in the Navy. The Army's 3.3b regulation calls for the Reduce Hazardous Components in Ordnance and Alternative Treatment for Hazardous Waste from Ordnance Processing. The Air Force instruction 974 requires Reduction of Lead Exposure at Firing Ranges.

Clean Air Act – lists lead compounds as Hazardous Air Pollutants that are subject to the National Emission Standards for Hazardous Air Pollutants rules (42 USC 7412(b)(1) – Jan. 2008).

National Ambient Air Quality Standard for lead was reduced to 0.15 mg/m^3. All facilities that 1 ton or more per year in urban areas (population great than 500,000) are required to install air-monitoring systems (40 CFR 50, 51, 58: 73 FR 66964 – Nov. 2008).

Occupational Safety and Health Administration (OSHA) the permissible exposure limit for lead compounds in the workplace at 50 $\mu g/m^3$ during an 8 hour shift (29 CFR 1910.1025 – July 2008).

National Institute for Occupational Safety and Health set an immediately dangerous to life and health value for metallic lead of 100 mg/m^3 and a recommended exposure limit for metallic lead, lead oxide, and lead salts of 50 $\mu g/m^3$ – Sept. 2005).
The American Conference of Governmental Industrial Hygienists established a threshold limit value for lead and inorganic lead compounds as 50 $\mu g/m^3$ and categorized inorganic lead compounds as Group 3A carcinogens.

Clean Water Act lists lead compounds as toxic pollutants (40 CFR 401.15 – July 2008). The National Pollutant Discharge System requires permits for the discharge of lead compounds into US waters (40 CFR 122 Appendix D – July 2008).

Safe Drinking Water Act set the maximum lead contamination goal of 0 μg/L in the National Primary Drinking Water Standard (40 CFR 141.51 – July 2008). Safe Drinking Water Act of 1986 lists lead compounds as carcinogens (27 CCR 27001 – Dec. 2008).

EPA began a Phase I assessment to determine if a 5 fold reduction in Soil Screen Levels is needed - 2010.

Resource Conservation and Recovery Act list lead and lead compounds as hazardous waste under codes F035-F38, K002, K003, K005, K046, K049, K051, K052, K061, K062, K064, K069, K086, K100, P110, and U144-U146. Lead styphnate is listed as a D003 reactive waste (40 CFR 261.3 – July 2008).

CERLA set the reportable quantity of lead nitrate (used to make lead styphnate) at 10 pounds.

The Toxic Substance Control Act requires lead, lead oxide, lead beta-resocylate and lead styphnate to be listed in the TSCA Inventory (15 USC Ch. 53 –Jan. 2007: CFR 710 – July 2008).

DOD Emerging Contaminant Watch List includes lead and a Phase I Risk Assessment on lead was completed in Jan. 2009.

2.0   DEMONSTRATION TECHNOLOGY

2.1   TECHNOLOGY DESCRIPTION

The current M52A3B1 primer is used for electrically primed 20 mm rounds in the M50 & M60 series and M197 Gatling guns by the DOD. The M52A3B1 primer uses the FA-874 Mix that contains lead styphnate as previously described in Table 1. In order to facilitate transition to production, our approach was to develop a material replacement for the FA-874 energetic material used to make M52A3B1 primers that would not require changes in the electric ignition system while providing similar or superior performance. In Figure 1, the primer cup and its components have been enlarged.

Figure 1: Conventional Primer Configuration
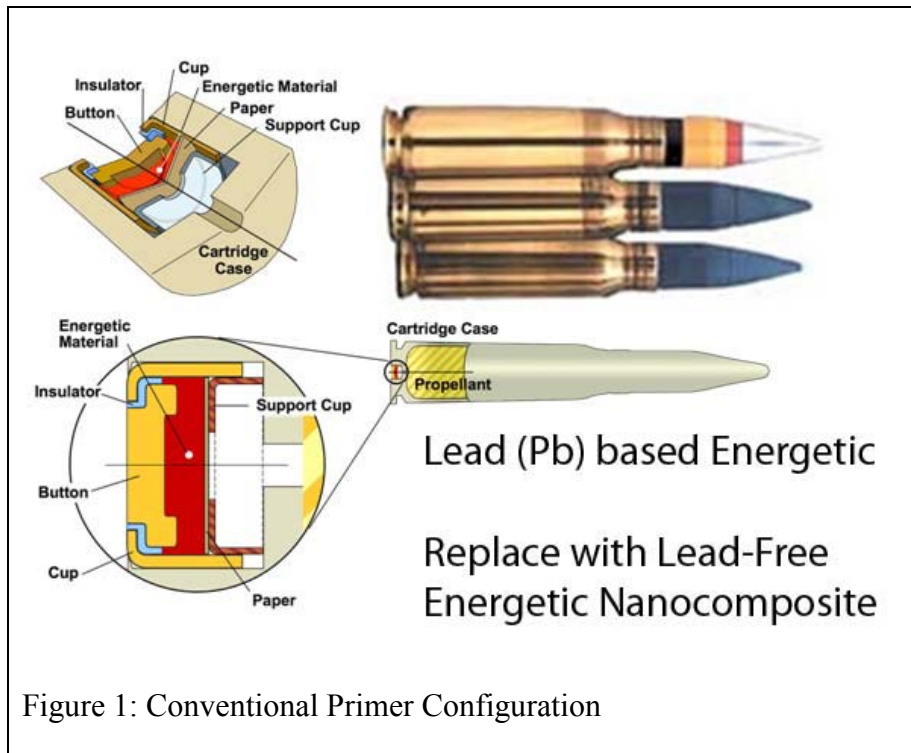
The primer cup is made up of a button that is insulated from the rest of the cup, energetic material (Mix FA-874) that is compacted in the cup above the button, a paper disk placed on the energetic material and a support cup placed on the paper disk. The primer cup is pressed into the cartridge case. The round is fired by the use of an electrical charge initiated at the button. Since the button is insulated, an electrical pathway must be established through the energetic material to the sidewalls. The electrical discharge between the button and the cup wall ignites the energetic material producing a hot stream of product that is pushed into the propellant bed, igniting the propellant. When a sufficient pressure is established by the burning propellant, the projectile is released. The AUR-AT measures the time from the ignition discharge until the projectile leaves the end of the barrel. The typical firing rate of 100 rounds per second for the M61 Vulcan Gatling gun is equivalent to one round fired every 10 milliseconds. To ensure gun safety, AUR-ATs of less than 3.5 ms is required.

The approach used in this ESTCP project was to develop a formulation based on an ENC to replace the Mix FA-874 containing lead styphnate. Once initiated, ENCs are known to react very rapidly, usually in 60 to 100 microseconds which is much faster than the 300 microsecond electric primer time cited in MIL-DTL-1394G Section 3.7. The electric primer time is the time from electric discharge to reach peak pressure for just the primer and should not be confused with the AUR-AT. The fast reaction rates of the ENCs make it ideal candidates as the energetic component of a lead-free primer. A wide variety of ENCs are known but only a few are suitable for primer applications. The main ENC used in the LFEP program is the Al/MoO_3 composite that has a heat of reaction (ΔH) of -4.70 kJ/g, -14.6 kJ/cc as compared to the lower ΔH for lead styphnate of -1.55 kJ/g, -7.02 kJ/cc. The energy density of the Al/MoO_3 composite is significantly higher than lead styphnate. A disadvantage of the Al/MoO_3 ENC is that it produces very little gas which is needed to propel the hot combustion products into the propellant bed. In order to achieve low AUR-ATs the LFEP formulation requires a gas additive to compensate for the low gas production from the ENC. The LFEP formulation also required a binder to enhance pellet integrity and firing reliability. Finally, carbon was added as a conductive additive just as the conventional M52 includes carbon to enable electric ignition.

The LFEP based on ENCs began with a SERDP Seed (WP-1183) where the feasibility was established. Originally, the LFEP were based on a LANL and NAWCWD joint patent by George Dixon, Joe Martin, and Don Thompson.[4] The SEED program demonstrated the need of a binder and gas additive to meet DOD's requirements. A follow-on SERDP (WP-1331) program led to the development of a LFEP composed of 71% Al(50nm)/MoO$_3$(45nm), 25% 3,6-bis(1H-1,2,3,4-tetrazol-5-amino)-s-tetrazine (BTATZ), 2% Kel-F® (polycholortrifluoroethylene) and 2% Carbon that met the DOD requirements at ambient and low-temperatures (LT). The ENC materials were made on a 1-to-2 gram scale by using ultrasonic mixing methods.

The overall technical approach for the follow-on LFEP program was to thoroughly investigate all aspects of ENC material for use in medium caliber ammunition primers from the perspectives of safety, reliability, production feasibility, cost, shelf life, and its ultimate performance in the intended application. For the purposes of this program, ENC was defined in its simplest form as a mixture of nano aluminum (Al) particles as the fuel and an appropriate oxidizer. The majority of the efforts associated with this program involved the use of molybdenum trioxide (MoO_3) as the oxidizing agent. However, in an effort to produce increased gas pressures and thereby improve primer performance, a number of different additives to the basic ENC material were evaluated.

To help ensure the successful accomplishment of program objectives, the Navy LFEP Team coordinated with other Navy labs as well as Army and DOE personnel from the Picatinny Arsenal and the LANL, respectively. There was successful collaboration in the evaluation of a number of different aspects of ENC formulations. In particular, the Navy LFEP Team benefited, because the Army shared knowledge gained through their investigation of the use of ENC for small-caliber percussion primer applications.

In addition to working with the China Lake LFEP Team, the LANL group also established independent working agreements with the NSWC-IH, to conduct analyses and tests in connection with Naval Sea System Command (NAVSEA) lead-free cartridge-actuated devices

and propellant-actuated devices. At the same time, LANL also supported the Army's work on the 25-mm M115 percussion primer. From the Navy's perspective, everyone benefited from sharing information about the characteristics of ENC material.

2.2   TECHNOLOGY DEVELOPMENT

Section 2 describes the technologies developed under the previous SERDP program.

2.2.1 Rapid Laboratory Nano Energetic Composite Assessment

In order to facilitate rapid evaluation of ENCs and primer formulations, the "Al Pan Dent Test" was developed under the ESTCP program.   In this test, a known quantity of ENC/primer is placed on a Disposable Aluminum Dish (Sigma-Aldrich Z154849), ignited with an Electro Technic Products, Inc Tesla Coil, the dent in the Al Pan measured, and the dent height divided by the weight of test material to give a "Deflection" in mm/g. Figure 2 shows the Al Pan Dent Test setup, the Al/MoO$_3$ ENC fireball, and the Al pans after ENC ignition.

Figure 2: Al Pan Dent Test

The Al/MoO$_3$ fireball was captured by high speed photography and the denting process occurred within the first 60 μs after initiation. These fast reaction times are needed for LFEP to meet the DOD AUR-ATs. The "Deflection" observed for the ENCs correlate well (Figure 3) to the peak pressures of ENCs in studies performed by Los Alamos in their pressure cells.

Figure 3 exhibits the Deflection versus the molar ratio of Al to MoO_3. Both LANL's pressure cell optimization and the Al Pan Dent Test gave an optimal Al/MoO_3 molar ratio of around 2.5 to 1. Later refinement gave an optimal ratio of 2.65 to 1. The Al Pan Dent Test enables easy fine tuning of the composition. LANL's method is time consuming and expensive.

Optimizations of Al/Bi_2O_3 and $Al/AgIO_3$ ENCs also correlated well.

Figure 3: Correlation of Al(80nm)/MoO_3 Optimization

2.2.2 Alternative Nano Energetic Composites (ENCs)

The base Al/MoO_3 ENC was selected in the previous SERDP program because it was the most studied system. However, there are a wide variety of thermite composites and many of the options are listed in "Theoretical Energy Release of Thermites, Intermetallics, and Combustible Metals".[14] Only a limited number of ENCs were characterized by the Al Pan Dent Test in the SERDP effort which summarized in Table 2:

Table 2: Al Pan Dent Test Deflections for ENCs

Fuel (Size)		Oxidizer (size)	F/O Wt Ratio	Fuel/Oxidzer Molar Ratio
Al(50 nm)	MoO_3(45 nm)	49/51	3.16	118
Al(80nm)	MoO_3(45 nm)	40/60	2.63	154
Al(80nm)	Bi_2O_3 (320 nm) Sigma-Aldrich	15/85	2.26	561

2.2.3 SERDP LFEP Composition

The final SERDP formulation was 76% Al(50nm)/MoO_3(45nm), 20% BTATZ, 2% Kel-F, 2%, Carbon Black and gave ambient and -65°F AUR-ATs of 2.98 $\pm$ 0.17 and 3.21 $\pm$ 0.12 ms, respectively.[15] The 50 nm aluminum vendor, Technanogy, went out of business and a preliminary test using 80 nm Al from NanoTechnology in place of 50 nm Al was evaluated. The primer gave poor results with ambient AUR-ATs in the 4-5 ms range.

2.3 ADVANTAGES AND LIMITATIONS OF THE TECHNOLOGY

The current LFEP based on ENCs have both advantages and disadvantages as compared to other

technologies. One significant advantage of the ENC based LFEP is that it is made by simple mixing and coating processes where overall LFEP yields are greater than 98%. Most alternatives require chemical reactions where yields can be low, product isolation is required and sometimes a specific crystal habit or size is desired thus requiring re-crystallization. Chemical synthesis also produces more chemical waste in the form of side-products, product salts, and solvents. In comparison, the solvents used in the preparation of the LFEP are recyclable without re-purification. A second general advantage is the ease of de-militarization of ENC based LFEP. These primers can be deactivated by a simple base hydrolysis that produces alumina, molybdate salts, ammonium nitrate (AN), and carbon so de-militarization cost will be low. A third advantage of ENC based LFEP is that they should be Hazards of Electromagnetic Radiation to Ordnance (HERO) safe.   The nanoscale dimensions of the composites are far too small to interact with millimeter and micrometer radiation wavelengths. This technology may enable the fleet to load ammunition without having to turn off high powered radar systems.   The major limitation is the reliability of vendors of nano materials. Due to economics, a vendor may or may not remain active in the production. Vendors require reliable customers with continuous product demand. Due to the fact that there are no deployed or commercial products requiring nano aluminum powders, the vendors have been unstable.   One promising application is nano Al-water propulsion that is being explored by the National Aeronautics and Space Administration (NASA). In the NASA program, 10 Kg of nano Al powder per month was ordered for 12 consecutive months. As additional applications develop, demand for nano aluminum powders should increase and help to stabilize the nano aluminum source. A second limitation is the fact that the environmental consequences of using nanoscale materials are generally unknown. Future environmental studies could lead to stricter government regulations that either prohibits the use or drives up the cost of utilizing nano materials.   Hopefully, future environmental regulations will be determined by scientific data and on a case by case basis. Close examination of the LFEP starting materials, the LFEP material and the LFEP reaction products by Scanning Electron Microscopy (SEM) shows that very little of the materials are loose nano particles. The LFEP and starting materials are highly agglomerated into multi-micron structures. Even simple dry mixing of nano aluminum and molybdenum trioxide forms self-assembling spherical agglomerates of 5 to 25 microns.   The LFEP reaction products are also mainly large micron particles and the loose nanoparticles was found to be only 0.2% of the product.   The products consisted of alumina, molybdenum metal, molybdates, molybdenum nitride and aluminum fluoride.

There are currently a number of materials that have been developed as lead-free replacements for lead-styphnate and lead azide.   The commercial ammunition industry has developed lead-free primers based on diazodinitrophenol (DDNP).[16,17] However, a US Air Force study raised questions about misfire rates, peak pressure consistency, ignition delays, and shelf-life.[18] The low temperature all-up round action times have also been an issue with the DDNP primers. Red-Phosphorous based primers were once used by the DOD in the late 1940's and several red phosphorus based primer compositions have been patented.[19,20,21,22]   While red phosphorus is more stable than white phosphorus, during storage it may release phosphine (PH_3) and phosphoric acids on exposure to oxygen and water. Due to the high toxicity of phosphine, OSHA permissible exposure limit is only 0.3 ppm.   A variety of encapsulation agents have been used to reduce phosphine emissions including epoxy or phenolic resins, rubbers, and aluminum hydroxide.   However, lead-free primers based on red phosphorus continue to be a liability because of the potential for phosphine release during storage and the de-militarization process. The P4 Primer™ developed by ATK has not been demonstrated for use in medium caliber ammunition and appears to have a 0.3 ms ignition delay for the 5.56 mm round as compared to the #41 Primer. If the ignition delay is inherent in the P4 Primer, it will not meet the 2.5 ms

primer action time needed for a M52A1B3 primer replacement (MIL-DTL-1394G Section 3.7). Copper 5-Nitrotetrazole also known as DBX-1 has recently been suggested as an alternative to lead azide. The variable crystallization induction period leading to variable crystal sizes appears to be a problem for scale-up. The fact that DBX-1 is copper based may be an environmental issue, especially in Sweden, Norway and the Netherlands where copper contamination in the aquatic environment is a problem. Even California considered banning (SB 623) copper based anti-fouling paints to prevent aquatic copper pollution. Copper based primers will introduce both air- and water-borne copper contamination into environment and could result in serious ramifications for the US Navy. DBX-1 has not been considered for use as a primer to date. Its main intended application is for lead-free detonators replacing lead azide. In the late 1990's, the potassium salt of 4, 6-dintro-7-hydroxybenzofuroxan (KDNP) was identified by Pacific Scientific Energetic Materials Company and NSWC-IH, as a potential replacement for lead styphnate. KDNP passed the NAVSEAINST 8020.5C material qualification testing as a primary explosive and has been evaluated in several applications including the CCU-63 Impulse Cartridge, Tube-launched Optically-tracked Wire-guided missile Initiator Units, and PVU-12/A Percussion Primers. Scale-up of KDNP was funded by Manufacturing Technology in 2010. Scale-up plants are planned for application for small caliber ammunition. Apart from the LFEP, KDNP appears to be the most viable alternative to lead-styphnate however it has not been demonstrated for use in medium caliber ammunition to date. The highest risk is that the low temperature all-up round action time will exceed 3.5 ms (MIL-DTL-1394G Section 4.3.6).

3.0   PERFORMANCE OBJECTIVES

The primers under development in this program are intended for electrically initiated medium caliber ammunition to be used by the US Army, Navy, Marines and Air Force. These automatic gun systems are designed for high firing rates up to 6000 rounds per minute. The primers are expected to meet the performance requirements of the Military Specification for the M52A3B1 Primer, MIL-DTL-1394G, and therefore provide a direct substitute. Table 3 contains the quantitative and qualitative objectives for the ESTCP LFEP project. The objectives were based on tasks that needed to be accomplished in order to have a product that could be transitioned into the DOD.

Table 3: Performance Objectives

Performance Objective	Primary Performance Criteria	Success Criteria	Results
Quantitative Performance Objectives			
Materials, Characterization, LFEP and 20 mm round build-up.	1. Characterize materials and determine requirements. Develop rapid characterization methods.	1. Characterization methods & properties identified for each starting material.	1. A list of characterization methods for each starting material was developed.
	2. Develop LFEP formulation.	2. Optimize a formulation that is lead and barium free with an AUR-AT of less than 3.5 ms.	2. A LFEP based on 80 nm Al, MoO_3, AN, binder and carbon was optimized.
	3. Develop methods to scale-up ENC and LFEP.	3. Develop processes to scale-up ENCs and LFEP.	3. A new scale-up method for NES developed & a patent issued. A scale-up process for the LFEP developed.
	4. Establish loading & consolidation methodology.	4. Loading & consolidation methods produce good AUR-AT data.	4. The hexane loading & consolidation using the original die set design was optimal.
Field Testing	5. Maintain specifications for basic materials and processes	1. Pass individual product tests described in the Military Specification, MIL-DTL-	1. Small scale LFEP samples showed reaction times of 60-100 μs

		1394G Pass material characterization and acceptance tests. The average AUR-AT plus 4 σ must be less than 3.5 ms at firing temperatures between -65 to 165°F.	well below the 300 µs criteria and the AUR-ATs averaged 3.04 ± 0.03 ms. The average -65°F AUR-ATs was 3.14 ± 0.09 ms. Full scale test in an autogun is pending.
	6. Eliminate lead (Pb) and barium (Ba) used in medium caliber electric primers	2. Satisfactory performance without identified hazardous materials	2. No lead or barium used in LFEP composition.
Qualitative Field Performance	1. Remove hazardous materials & reaction products w/o affecting system performance	1. Less hazardous materials released to the environment	1. Pb and Ba removed but the hazards of nano products are still unknown.
	2. Maintain military field environmental requirements	2. No effect on utility or shelf life of product	2. Full aging study not complete. Samples aged for > 5 years still function.
	3. No impact to manufacturing techniques	3. Similar industry process controls	3. Water loading is not possible. However, a suitable fluorocarbon solvent with low global warming potential could be used in place of water. A solvent recycling system would be needed to reduce cost.

Quantitative: The first criterion for the starting materials is the identification of suitable starting material, determining which characterization methods needed for each material, and the properties required for each material. For the nano aluminum powder fuel, the active aluminum content, the nano Al/oxidizer molar ratio, particle size and particle size distribution are critical for high performance in the LFEP formulation. The oxidizer selected has a major impact on the behavior of the ENC and LFEP. MoO_3 (45nm) was selected but other oxidizers have been identified as being as good. Low-density ammonium nitrate from Dyno Nobel was selected. In

the Al Pan Dent Test, Dyno Nobel produced the best deflections. It is unclear as to why but each manufacturer uses different phase stabilizers that are company proprietary information. The binder selected was Kel-F which is a PCTFE. This material has different names depending on vendor including Halocarbon 600, Neoflon, Aclar and PCTFE. The main criterion for the PCTFE binder is having a melting point above 65°C.

The second qualitative materials criterion was to develop a functional primer material since the SERDP developed LFEP was not suitable. A LFEP was developed based on 80 nm Al (versus 50nm), MoO_3, AN (versus BTATZ), Kel-F and Carbon Black. The fuel-oxidizer ratio and the amount of gas additive were optimized.

The third performance criterion was the development of scale-up methods that did not detrimentally impact performance. Scale-up methods for ENCs did not exist and this was a high risk element of the program. Scaling up the LFEP was also a risk but primary safety and performance risks. Preparing materials on large scales meant an accidental mishap could be life threatening. Most steps in the scale-up procedures were achieved in solvents and the safety risks mitigated. As with most scale-up efforts, the impact is not known until final testing. The scale-up of the ENC was outstanding. The deflection number for the scale-up samples usually exceeded those from the small scale preparations. The scale-up of the LFEP was slightly poorer than the small scale but this was probably due to poorer mixing in the final carbon addition step. Longer mixing time should improve the uniformity of the material.

The fourth materials criterion was the loading and consolidation of the LFEP into primer cups. The optimal loading conditions for performance needed to be determined, a method was required to increase loading speed and the multi-die set had to be evaluated. Wet hexane loading using the original die set design produced the best AUR-ATs. The loading rate of primers could be dramatically increased with the use of a powder injector system but the AUR-ATs increased due to some low loading levels. The multi-die set produced reasonable results but increased the manual labor required and slowed down the consolidation process.

Criterion 5 of maintaining the performance specifications of the M52A3B1 primer is the most critical metric. With the high firing rates used by the DOD, a long AUR-AT could result in a hang fire where the 20 mm round is initiated but does not fire until the rotating gun barrel is misaligned with the chamber resulting in gun damage. The maximum firing rate of 7200 rounds per minute for the M61A2 Gatling gun requires the AUR-AT to be 3.5 ms or less. The MIL-DTL-1394G Table III requires an average primer time (just the primer not the all-up round) below 300 μs $\pm$ 3σ. The LFEPs have an average primer time of 80 $\pm$ 20 μs. In addition, the All-Up Round Action Time plus 4σ will not exceed 3.5 ms at -65°F. The final down selected LFEP composition resulted in AUR-ATs of 3.12 $\pm$ 0.08 ms meeting the MIL-DTL-1394G requirement.

Criterion 6 eliminating lead and barium from the M52 primer is fully achieved in the LFEP. Neither lead nor barium is used.

Qualitative Field Testing Criteria: Criterion 1 is the removal of hazardous materials & hazardous products. Airborne lead and barium will were eliminated making the testing environment safer. However, the LFEP contains nanomaterials whose hazard properties are not been fully determined and is a subject of current research. The fate and effect of nanomaterials in the environment are currently unknown but the nanomaterial in a 20 mm round is very small

accounting for < 0.3% of the primer plus propellant weight. Product studies of the pure Al/MoO$_3$ ENC and LFEP showed that < 2% was smaller than 100 nm of which 90% was trapped on the surface of larger 2-50 micron particles. Thus less than 0.3 mg of nanosized product is released per 20 mm round. Criterion 2 is having a long shelf life without performance loss. This study has not been completed. An aging study was started on the LFEP prepared by sonication method by NSWC-Crane but the study was terminated after 6 months. PMA 242 wanted the study done on the scaled-up LFEP material. Criterion 3 is that the LFEP would have no impact on the manufacturing process. The manufacturing of the LFEP material is very simple compared to the complex series of chemical reaction steps used to make the Mix FA-874. The preparation of the LFEP material is a series of mixing step where there is little to no yield losses. In order to replace the Mix FA-874 with the LFEP, Lake City would have to replace the use of water with a fluorocarbon solvent in their loading and consolidation process. The use of water is not possible for the ESTCP LFEP formulation. The loading and pressing was successfully demonstrated using fluorocarbon solvents. A fluorocarbon solvent with a very low vapor pressure and low global warming potential could replace water but a solvent recycling system would be needed for cost consideration issues.

The viability of the ESTCP LFEP has been demonstrated on a small scale. The LFEPs met the performance metrics in MIL-DTL-1394G.

4.0 SITE / PLATFORM DESCRIPTION

4.1 TEST PLATFORMS/FACITIES HISTORY

The Test Site is located at the NAWCWD/CL China Lake, California. Investigations and material characterizations take place in the Michelson Laboratory and the Center's Ballistic Test Range both manned by senior research personnel and accomplished Test Range personnel skilled in gun systems and ammunition testing.

Ordnance Development and Testing has been conducted by dedicated Scientists and Engineers since the research center was established in 1943 and has evolved into the Navy's premier Research and Development Center responsible for the development of major weapons and weapon systems used by the US military and Armed Forces around the world.

The Picatinny Arsenal Armament Technology Facility is a full-service design and development laboratory for small and medium caliber weapon systems. Picatinny has 4 weapons validation bays with environmental chambers capable of controlling temperatures between -65 and +165F where guns and ammunition can be conditioned and fired. Data acquisition capabilities include a high speed camera with shutter speeds up to 150,000 frames per second.

4.2 PRESENT OPERATIONS

A description of the LCAAP preparation of the Mix FA-874 and loading of primers for 20 mm rounds was described in Section 1.2. The 20 mm ammunition addressed in this project is fired by M50/M60 and M197 Gatling guns over and on DOD ranges all over the country in testing and training exercises from aircraft such as FA/18s, helicopters, and by Phalanxes.

4.3 SITE-RELATED PERMITS AND REGULAIONS

Hazardous Material Handling

Energetic Material Storage

Health and Safety Standard Operating Procedures (Appendix D)

IAW Navy explosive safety regulations

5.0 TEST DESIGN

5.1   MATERIALS & CHARACTERIZATION

The materials characterization is an extremely important component in the performance of LFEP based on energetic nanocomposites. Changes in average particle size, particle size distribution and purity can dramatically change the performance of the LFEP. Starting materials from commercial vendors have been proven to be unreliable. Vendor's claims on particle size, active aluminum content, and purity are often wrong. Chemical materials from different vendors are often quite different varying in average particle size, morphology, and crystal habit. In addition, lot-to-lot variance of materials from a single vendor is a persistent problem. Manufacturers change their process impacting their product performance and do not notify customers of the changes. At this point in time, independent characterization of starting materials is essential. To determine the amount of variance that is acceptable for production purposes materials were ordered from different vendors and multiple lots were obtained from single vendors. The impact of product variance on LFEP performance is an important issue. Some materials also age with time and the aging process affects the chemical properties that impact performance. Vendors of a material sometimes modify the material with proprietary coatings that can also impact primer performance. This leads to variations in primer performance due to differences in materials from different vendors.

5.1.1 Materials

The major materials used in this program and the vendor sources are listed below:

Al (50 and 80 nm) – Technanogy, Inc/NovaCentrix
ALEX (Al (150 nm) Exploded aluminum powder) – Argonide Corporation
Flake Al (150 nm thick by 2 microns diameter) – Toyal
Bi_2O Bismuth Trioxide – Nanophase (50 nm Bi-0250EXP), Aldrich (320 nm 637017), Skylighter (1.5 and 2.5 micron CH8040), Sigma-Aldrich (10 micron #223891), Atlantic Engineering (10 micron).
Carbon Black – Chevron-Phillips
Kel-F (PCTFE) - The Kel-F® brand (Polychloro Trifluoroethylene) is a registered trademark of 3M, but they discontinued production in 1995. Neoflon ® (PCTFE) is made by Daikin Industries of Japan. Analogous materials are sold by Halocarbon Corporation Halocarbon 600.
MoO_3 (45 nm EM-NTO U2) – Climax Molybdenum
$AgIO_3$ NAWCWD (Submicron), City Chemicals (1.6 micron), and Noah Chemical (1.75 micron)
NH_4NO_3 (AN)
   - Low Density: AN Baker, Wickman, Dyno Nobel
   - High Density: Sigma-Aldrich

5.1.2 Material Characterizations

While each starting material could be characterized by every technique it would be inefficient since some characterization techniques do not reflect on its performance. Specific sets of characterizations were developed for each material. Section 5.2.2 includes brief summaries of material characterization methods for starting materials and composites. These characterizations are essential to assure that starting materials are consistent from batch to batch.

5.1.2.1 Thermogravimetric Analysis (TGA) - The TGAs were performed on a TA Instruments 2950 thermogravimetric analyzer with a DuPont 2100 thermal analyst controller. Sample weights were between 1 to 5 mg and data were corrected for instrumental drift using a blank. The standard TGA experiment for aluminum samples was to ramp up the temperature from room temperature to 350°C at 20°/minute, slow the heat rate to 3°/minute between 350 and 600°C, heat at 20°/minute to 850°C and hold for 4 hours. Under these conditions, Al particles larger than 500 nm did not fully oxidize leaving behind a gray residue.

5.1.2.2 Surface Area Measurement - The surface area of the nano and micron powders were also determined by Brunauer, Emmett and Teller (BET) using Quanta-Chrome Autosorb-1C surface area analyzer. Samples were heated under vacuum to 250°C for 24 hours to remove adsorbed gases. The samples were cooled to -196°C and the nitrogen adsorption of nitrogen obtained. The surface area was calculated using the BET equation.

5.1.2.3 Scanning Electron Microscopy (SEM) - The SEMs were collected on an Electroscan Environmental Model E-3 and an Amray Model 1400 to examine the particle size, particle morphology and agglomerization.

5.1.2.4 Electrostatic Discharge (ESD) – An Electrostatic Discharge test system was developed in-house to evaluate highly ESD sensitive materials. Standard Safety testing has a lower limit of 250 mJ. NAWCWD's in-house ESD Tester can detect sensitivities down to 4 µJ and up to 2.5 J. The ESD Tester is shown in Figure 3:
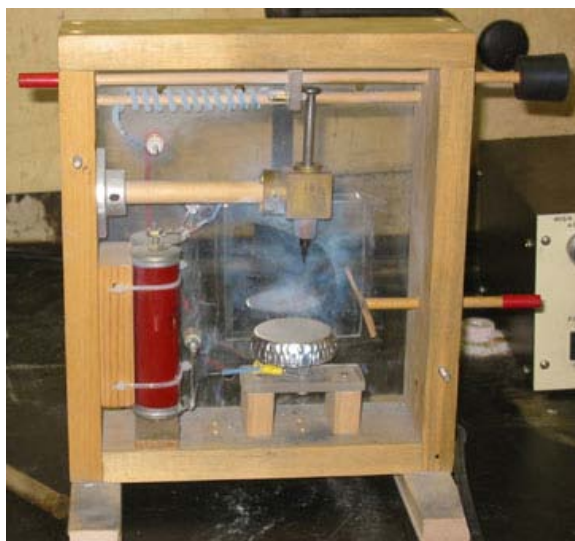
Figure 3: NAWCWD ENC ESD Tester

The spark energy (Esp) is defined by the equation. 2 below:

$$Esp = \frac{1}{2} CV^2 \text{ (J)} \qquad (2)$$

C = Capacitance (µJ)
V = Voltage (KV)

The capacitance of the system is set by two 0.01 µJ capacitors linked in series for a total capacitance of 0.02 µJ. The voltage is manually set on a Betran Associates Inc. High Voltage Power Supply Series 230 with a range of 0.01 to 5.0 KV. The spark gap is set by an adjustable spinning base that raises and

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lowers the base electrode platform. For very sensitive materials the gap was minimized by attaching a Volt-Ohm meter to the 2 electrodes and minimizing the gap while maintaining a resistance of 20 M-Ohm. The gap for less sensitive materials was set at 0.002 inches. The onset energy (E_{onset}) is the lowest amount of energy required for ignition of a material. The 50% point ($E_{50\%}$) was determined with at least 5 measurements where half or more of the samples tested ignited. The ESD data is listed in Appendix I.

5.1.2.5 Base-Hydrolysis - The TGA and base-hydrolysis were performed to determine the active aluminum (or metallic) content. The base-hydrolysis was conducted on ~0.2g Al samples using a 1M NaOH or KOH solution. The hydrogen gas was captured, volume measured and the active aluminum content calculated based on the stoichiometric reaction in Eq. 3:

$$2 \; Al + 2 \; MOH \; (M = Na, K) + 6 \; H_2O \rightarrow 2 \; MAl(OH)_4 + 3 \; H_2 \quad\quad\quad (3)$$

The base-hydrolysis determines the actual active aluminum content while the TGA using our protocols determines the amount of aluminum that oxidizes below 850°C.

5.1.2.6 Al Pan Dent Test - Initially, ENCs were prepared based on LANL's optimized compositions. NAWCWD had no means to measure the performance of the ENCs and so alternative ENCs could be made but not optimized or adequately compared to Al/MoO_3. To address this issue, a convenient rapid laboratory test method was developed to facilitate the development of ENCs and the LFEP material. The **Al Pan Dent Test** was originally developed at NAWCWD for characterizing ENC in the SERDP project "Lead-Free Electric Primer" WP-1331. The concept is similar to the witness plate test for explosives developed by LLNL. However, only a few ENCs had been characterized by the Al Pan Dent Test in the SERDP effort. A complete description of the test, critical parameters and procedures are found in Appendix B. **Personal Protective Equipment should include a grounding wrist strap, safety glasses, hearing protection, and laboratory coat.** The Al Pan Dent Test setup is shown in Figure 4:
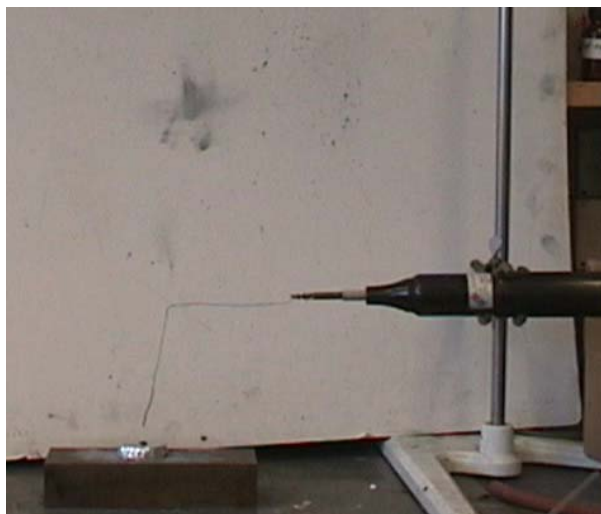
Figure 4: Al Pan Dent Test Setup

The Al Pan Dent Test is performed by placing a small amount of ENC on a Sigma-Aldrich Disposable Aluminum Dish (Z154849). The amount depends on the ENC. For $Al(80nm)/MoO_3(45nm)$, $Al(80nm)/Bi_2O_3$, and $Al(80nm)/AgIO_3$, the standard amount are 18, 12, and 6 mg, respectively. An effort was made to produce a clean dent without distorting the pan sidewalls or penetrating the Al pan. The ENCs were broken down into fine powders with a grounded spatula. The inclusion of chunks of ENC will result in low deflection numbers. The Al Pan is weighed on a grounded balance, the ENC placed and piled (small as possible) on the center of the pan, the Al pan + sample weighed to determine the amount of ENC.

The sample is placed under a wire attached to the tip of an Electro Technic Products, Inc Model BD-10A Tesla Coil. The Tesla Coil creates a spark that initiates the ENC and creates a dent in the Al pan. Example of a dent and hole in the Al Pans are shown in Figure 5:

	The denting process was investigated by high speed video under a Defense Threat Reduction Agency (Grant/MIPR #10-2954M Dr. Suhithi Peiris, Dr. William Wilson) and found to form within the first 60-100 μs.
Figure 5: Al Pans From Testing of 25 mg of Al(80 nm)/MoO$_3$(45 nm) (left) and 13 mg of Al(80nm)/AgIO$_3$(270 nm) ENC	
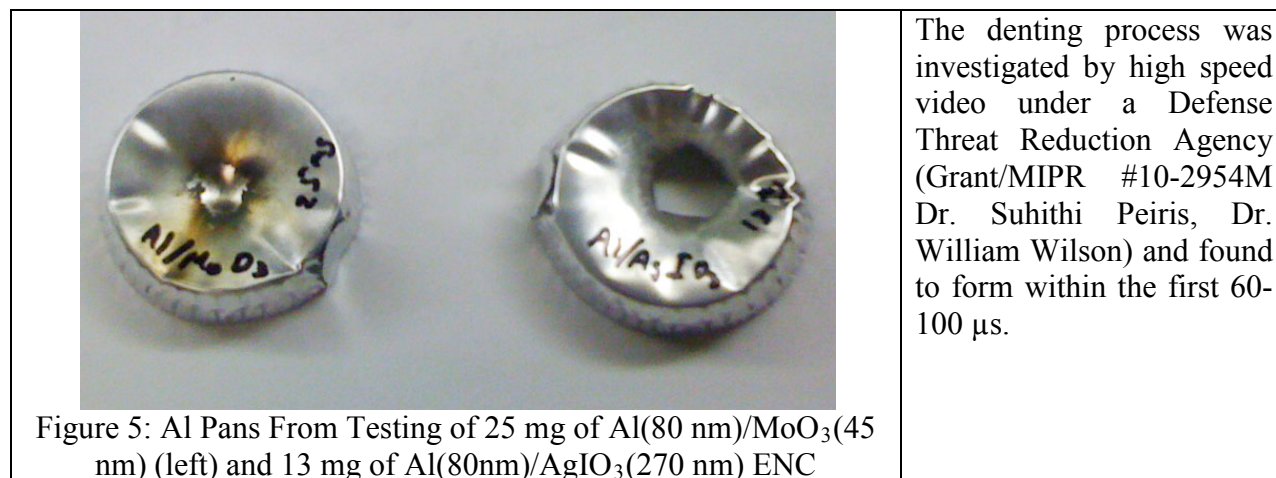
In Figure 5, 25mg of Al(80 nm)/MoO$_3$(45 nm) (left) and 13 mg of Al(80 nm)/AgIO$_3$(270 nm) (right) were ignited with a spark producing a dent with a Deflection of 154 mm/g and a hole. The ENC of the left is currently used in the LFEP and the material on the right is the backup ENC for LFEPs.   The dent is measured by placing the Al pan on a flat Al plate. The height of the dent plus Al plate plus Al pan is measured with a Scherr-Tumico micrometer. The dent height is calculated by subtracting out the Al plate and Al pan thicknesses. The Deflection (mm/g) is then calculated by dividing the dent height by the weight of the test sample. When a hole is formed, the amount of material tested is reduced until only a dent is formed without crinkling of the Al Pan sidewalls.

The Al Pan Dent Test is one of the most important technologies developed in the LFEP ESTCP program because it enables:
    1) Rapid optimization of Al(80 nm)/MoO$_3$ with new nano Al batches
    2) Rapid evaluation and development of alternative ENCs
    3) Evaluating alternative gas additives
    4) Evaluation of the effects of additives and additive loading levels
    5) Evaluate how process changes affect the material performance
    6) Check LFEP consisty from batch to batch
    7) Check the quality of new batches of staring materials without having to do AUR testing
    8) Assessing the scaled-up process and batches before AUR testing
    9) Identifying critical factors affecting AUR-ATs
    10) Provides the Industry with a rapid process for quality control and assessment.

5.1.3 LFEP Loading/Consolidation

Some different loading and consolidation parameters were investigated in this program. Some

potentially critical parameters for LFEP loading included weight of primer (140-170 mg), dry versus wet loading, solvent used in wet loading, weight variance when using the powder injector, post press drying step, press cleaning and lubricating materials.   These parameters were investigated using the AUR-AT as the basis for determining their importance.   Although Dry Loading of the primer cups was our standard method, wet loading using solvents (hexane and iso-propanol) was investigated to reduce risk of accidental ignition during the loading process. It was important to determine if the wet loading process impacted the all-up round action. Some potentially critical consolidation parameters identified include consolidation pressure, die configuration, multi-die configuration, and location effects when using the multi-die set on AUR-AT (center, edges, and corners).   Pressing procedures were done in accordance with Standard Operating Procedure SOP 10090-03, "Pressing of Energetic Material".   Sections 5.3.1, 5.3.2, and 5.2.3 describe three different LFEP loading processes tested.

5.1.3.1 STANDARD LFEP WET LOADING OPERATION (Single Die)
Wet primer mix charge with solvent (Hexane/Iso-Propanol) and stir to mix to a uniform slurry
Insert primer cup in die holder and spoon in slurry.   (Primer cup has insulator installed) Scrape any residue particles into cup
Insert ram punch into die set – Large End
Single die set with ram punch is placed in Press. Consolidate primer mix at 300 psi
Remove primer cup from die set
Repeat process for each Primer Cup.
Pressed mix and primer cups are dried 4 hours @ temperature (51°C)
Insert cup with dried primer mix into die set
Insert primer-disc over primer mix
Insert support cup, flange side up
Insert ram punch into die set – Small End
Die set placed in press and consolidated at 8000 psi
Remove die set and remove finished primer
Place finished primer in handling/storage container.
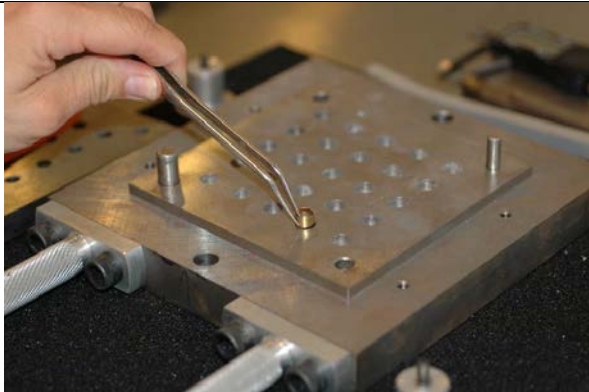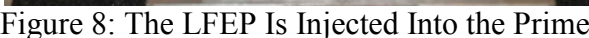
5.1.3.2 LFEP DRY LOADING OPERATION (Single Die)
Insert primer cup in die holder and spoon in LFEP
Insert cup with dried primer mix into die set
Insert primer-disc over primer mix
Insert support cup, flange side up
Insert ram punch into die set – Small End
Die set placed in press and consolidated at 8000 psi
Remove die set and remove finished primer
Place finished primer in handling/storage container.

5.1.3.3 LFEP DRY MULTI-DIE LOADING OPERATION

In order to increase primer consolidation throughput, a 5x5 multi-die set was fabricated. This system would enable 25 primers to be pressed simultaneously instead of one at a time. The design of the multi-die-set enabled tracking of each of the 25 primers so that edge and corner effects could be monitored. It is important that the multi-die set does not lead to variances in the all-up round action times or misfires. The consolidation pressures at the edges and corners of the 5x5 matrix may be different than the center. The multi-die set loading process is shown in Figures 6 to 13:

 Figure 7: Cups Are Placed in the 5x5 Multi-die Set	In Figure 6, the primer cups are placed into the 25 holes in the multi-die set. The two different alignment pins distinguishes the orientation of the multi-die set and enables tracking of the individual primers. The different sized alignment pins also ensure that the press stack can be assembled in only one way.
 Figure 7: The Powder Injector Is Loaded with 165 mg of LFEP	In Figure 7, the LFEP powder is drawn into a commercially available powder injector. The powder injector system was grounded to prevent accidental ignition of the LFEP. The powder injector system is tunable and it was set to pick up 165 mg of LFEP.
 Figure 8: The LFEP Is Injected Into the Primer Cup	In Figure 8, the LFEP powder is loaded into the primer cups. The variation in weight was found to be < 2 mg.
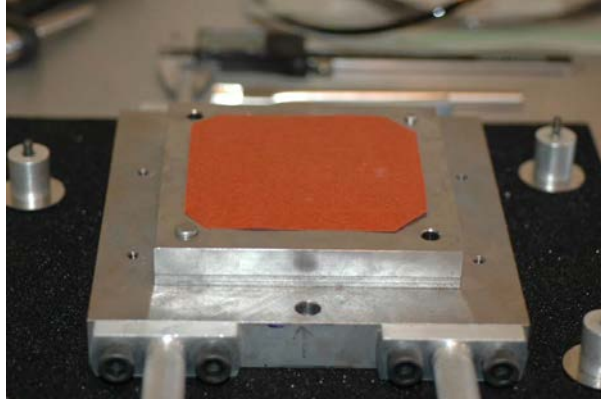
Figure 9: The Seal Paper Is Placed Over the Cups

In Figure 9, the Seal paper is placed over the 5x5 matrix of LFEP cups.

Figure 10: The Support Cup Holder Is Put In Place

In Figure 10, the Support Cup Holder plate is placed on the Seal paper using the alignment pins.
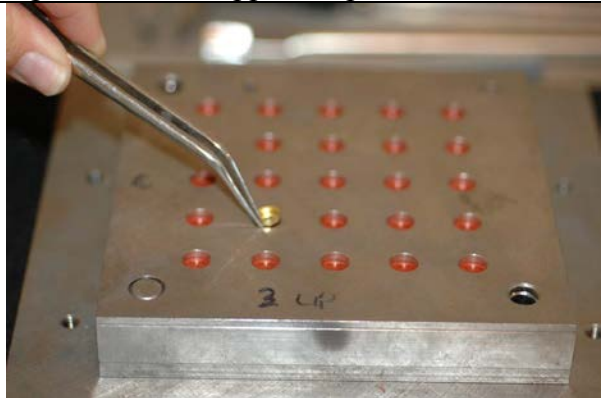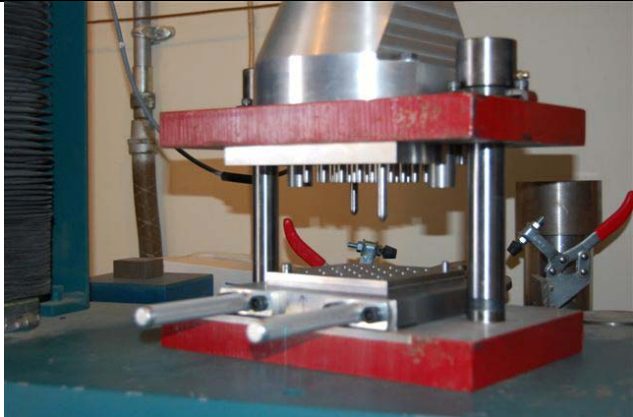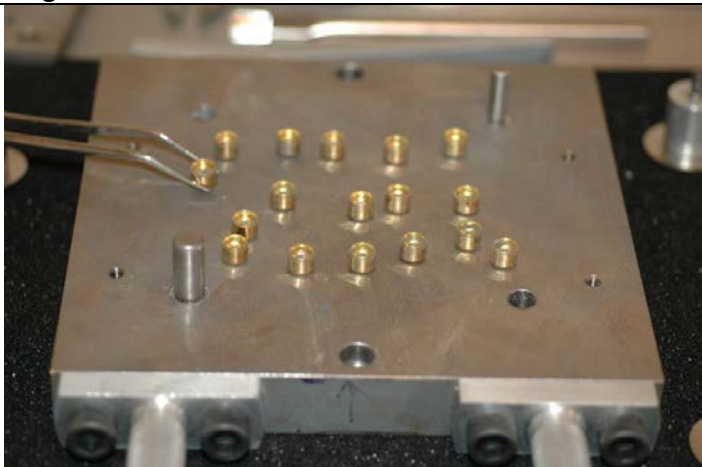
Figure 11: Insert Support Cups

In Figure 11, the 25 Support Cups are placed in the Support Cup Holder plate. The plate aligns the Support Cups with the Primer Cups in the lower plate.

	In Figure, 12, the multi-die set is transferred to the press where the top plate of the multi-die set is inserted using alignment pins. The 25 primers are pressed at 7500 PSI simultaneously.
Figure 12: Die Set Placed In Press and Consolidated at 7500 psi	
	In Figure 13, the multi-die set is disassembled and the primer cups pressed out of the primer cup holder plate. The primer cups are placed in a tray with numbered holes for primer cup tracking.
Figure 13: Remove Die Set and Remove Finished Primer	

5.2   FIELD ALL-UP ROUND 20 MM AMMUNITION TESTING

Demonstration functional testing was performed at the Ballistic Test Range which is equipped with modern instrumentation for parameter characterization.   The 20 mm round testing was conducted in an outdoor range with a target berm capable of trapping medium caliber ammunition. An instrumented Mann Barrel gun was used for the Pressure (barrel), Velocity (projectile), and Action Time measurements where 20 mm rounds with conventional M52A3B1 primers were used as the baseline. The conventional primed rounds generate a barrel pressure of 50,000 psi, a projectile velocity of 3,380 feet per second, and ambient/-65°F AUR-AT of 2.90/2.95 ms.   The intended use of the LFEP are for the M61A2 Gatling gun used in aircraft such as the F/A-18 and F-22 and the M197 Gatling gun used in DOD helicopters. The M61A2 Vulcan Gatling Gun used in the F/A-18 is capable of firing a rate up to 7200 rounds per minute (rpm). However, it is switch configured to fire at either 4000 or 6000 rpm and hang fire conditions occur at AUR-ATs of 8 and 5 ms, respectively. Under hang fire conditions, the round is electrically initiated, the barrel over rotate before the round fires and the round damages the gun. This is avoided by having a low AUR-AT.

In the SERDP program, the Mann Barrel gun was used for PVAT testing at ambient and low temperature. For low temperature firing, the ammunition was cooled to -65°F for 4 hrs and the

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breach/chamber cooled to 0°F. With external temperatures exceeding 115°F during the summer, the actual firing temperature was highly variable. To achieve consistent firing temperatures, the entire Mann Barrel was wrapped with copper liquid nitrogen cooling coils and placed in a wooden shroud 14. The gun was cooled to -65°F, the conditioned 20 mm round loaded and fired. Due to the large gun mass, the temperature of the gun increased by about 10° per hour at 110°F. The temperature was regulated by periodic liquid nitrogen flow.
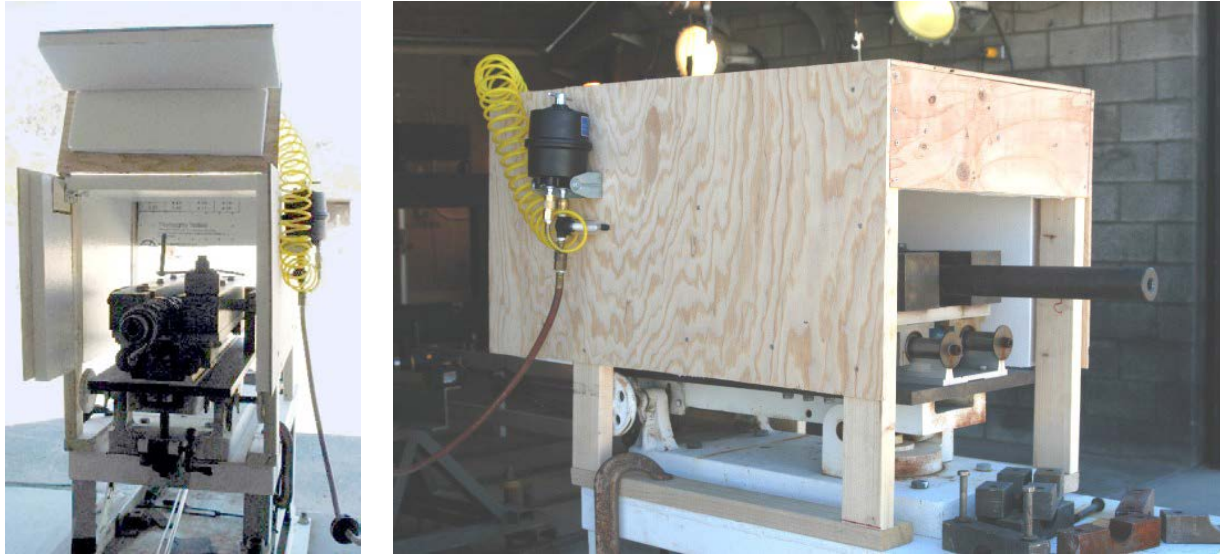
Figure 14: Low Temperature Setup for 20 mm All-Up Round Firing

The barrel pressure, the projectile velocity and action times were measured using the Mann Barrel at ambient and low temperature. The normal 20 mm round contained a primer weight of 178 mg of Mix FA-874 (used in M52A3B1 primers), 580 grains of WC 868 propellant, and a 100 g projectile.

6.0 Performance Assessment

At the end of the SERDP program, a suitable LFEP composition was identified but the Vendor for the 50 nm Al discontinued their business, the gas additive, BTATZ, was still not commercially available, and the binder, Kel-F, was made solely in Japan. This required a major re-development of the LFEP formulation, characterization of new materials, and re-optimization. In addition, methods for the production of ENCs in large scales were unknown. If a scale-up method could be developed, the materials had to be characterized and compared to the ENCs prepared by ultra-sonication methods. The LFEP is comprised of 5 components, a fuel, an oxidizer, a gas additive, a binder and a conductive additive for electric ignition. A gas additive to replace BTATZ and a replacement for Kel-F had to be identified and tested. The composition of the LFEP had to be optimized by varying the amounts of each component, testing the formulation in the laboratory, and ultimately performing full Mann Barrel Gun testing. Starting materials are often plagued with batch-to-batch variations so a rapid laboratory test was needed to screen starting materials. The effects of the variations on the AUR-ATs had to be characterized to determine critical parameters to ensure primer reliability. Section 6 describes the characterization of materials which is extremely important for primer reliability and reproducibility, the formulation optimization, scale-up, safety testing, and ammunition testing. The ultimate performance assessment tool was the ambient and low-temperature All-Up Round testing where barrel <u>P</u>ressure, projectile <u>V</u>elocity, and <u>A</u>ction <u>T</u>imes (PVAT) were measured.

6.1   MATERIALS AND CHARACTERIZATION

Although some of the starting materials were characterized in the previous SERDP, they had to be re-characterized in the ESTCP effort as well due to potential aging. The starting materials for the LFEP composites were characterized by a variety of techniques including Thermogravimetric Analysis (TGA), base-hydrolysis, Surface Area Analysis using Brunauer, Emmett, Teller (BET), Scanning Electron Microscopy (SEM), powder X-ray analysis, Fourier Transform Infrared (FTIR) Spectroscopy, and the Al Pan Dent Test. The TGA and base hydrolysis was used to determine the active (metallic) aluminum content in nano aluminum powders. In general, the hydrolysis of nano aluminum powders gave a higher active Al content than TGA. The base hydrolysis oxidized all of the aluminum while the TGA using NAWCWD's standard conditions completely oxidized only particles under 500 nm. The difference between the active Al content between TGA and base hydrolysis is due to large particles that reduce the nanopowder reactivity. The TGA and SEM were used to assess the Al particle size distribution.   The BET and the powder TGA were used to calculate average particle size. The powder X-ray analysis was used to determine purity and identify crystalline components in the starting materials. The FTIR was used to determine sample purity. Not every technique was used on each material and a recommended characterization list was generated for each class of starting material.

6.1.1 Aluminum/Fuel Powders – Three 80 nm Al batches were evaluated during the ESTCP effort, Technanogy's M2210 and Novacentrix's M2453, M2671 and M2697. ALEX (150 nm) powder was also investigated as an alternative to 80 nm Al.

6.1.1.1 Active Al Content. The Technanogy's M2210 80 nm Al sample was initially characterized in the SERDP program in 2005. The 80 nm Al powder aged over time, starting off at 87% active metal and dropping to 74% after 9 weeks in air. After more than 5 years in air, the active Al content was determined to be 74% by TGA and base hydrolysis. Virtually no additional aging occurred after the first 9 weeks of aging in air. The Novacentrix's M2671 gave an active Al content of 77.6% by TGA and 81.7% by base hydrolysis. The difference in active Al content is due to large Al particles (> 500 nm) in M2671 that do not oxidize in the TGA. The M2671 batch of 80 nm Al was made on NovaCentrix's production reactor that has less control over particle size distribution. The Novacentrix's M2697 that was made on the production reactor gave an active Al content of 70.9% by TGA and 74% by base hydrolysis. It has the same large particle problem as the M2671 sample. A valuable technique was developed at NAWCWD to calculate the approximate Al particle size distribution based on TGA data.[23] This technique can be used by NovaCentrix and other nano aluminum vendors to determine their particle size distribution from run-to-run and identify problems during production. Argonide's Exploded Aluminum (ALEX 150 nm) had an active Al content of 84.9%. The cost of ALEX was about 10% of the cost of 80 nm Al and has a higher active Al content. The substitution of ALEX for 80 nm Al powders would significantly reduce LFEP costs and was investigated in the ESTCP program.

6.1.1.2 Electrostatic Discharge Sensitivity of Al Powders. The ESD sensitivity of nano Al powders was examined using our in-house built ESD Tester. A positive or Fire condition occurs when the spark ignites the Al powder. A No-Fire is when the spark disperses the powder without an ignition and the results are summarized in Table 4:

Table 4: Electrostatic Discharge Sensitivity of Nano Aluminum Powders

Al Sample	Capacitance μF	Voltage (KV)			Energy (mJ)		
		No-Fire	E_{Onset}	$E_{50\% Point}$	No-Fire	E_{Onset}	$E_{50\% Point}$
Al(33nm)	0.02	0.50	1	1.25	2.5	10.0	15.6
Al(50nm)	0.02	0.25	0.5	0.5	0.6	2.5	2.5
Al(80nm)	0.02	1.00	1.25	1.5	10.0	15.6	22.5
ALEX(150nm)	0.02	1.00	1.25	1.5	10.0	15.6	22.5

The 50 nm Al powder is significantly more ESD sensitive than the other aluminum powders. The reason for this is unclear especially considering the 50 nm Al powder has aged in air for over 7 years. Usually, the ESD sensitivity decreases over time as the oxide layer thickens during

aging. The higher ESD sensitivity indicates the 50 nm Al is much more reactive than the 33, 80 and 150 nm Al powders. The high reactivity is needed for low AUR-ATs.

6.1.1.3 Optimization of 80 nm Al Powders Using the Al Pan Dent Test – To optimize different batches of 80 nm Al, a series of Al/MoO$_3$ composites were prepared by sonication methods where the weight percent of nano aluminum powder was varied between 25 and 75%. Specific amounts of ENC were placed on Al Pans and ignited. The deflection was calculated from the dent height divided by the weight of the sample. In general, the Al(80nm)/MoO$_3$ ENCs using different batches of 80 nm Al powders from NovaCentrix and Technanogy optimized at nearly the same nanoaluminum weight percentage (40 $\pm$ 0.5%) with a maximum Deflection between 154 to 164 mm/g. The optimization of Al(80 nm)/MoO$_3$ composite using several batches of 80 nm Al powders is shown in Figure 15.
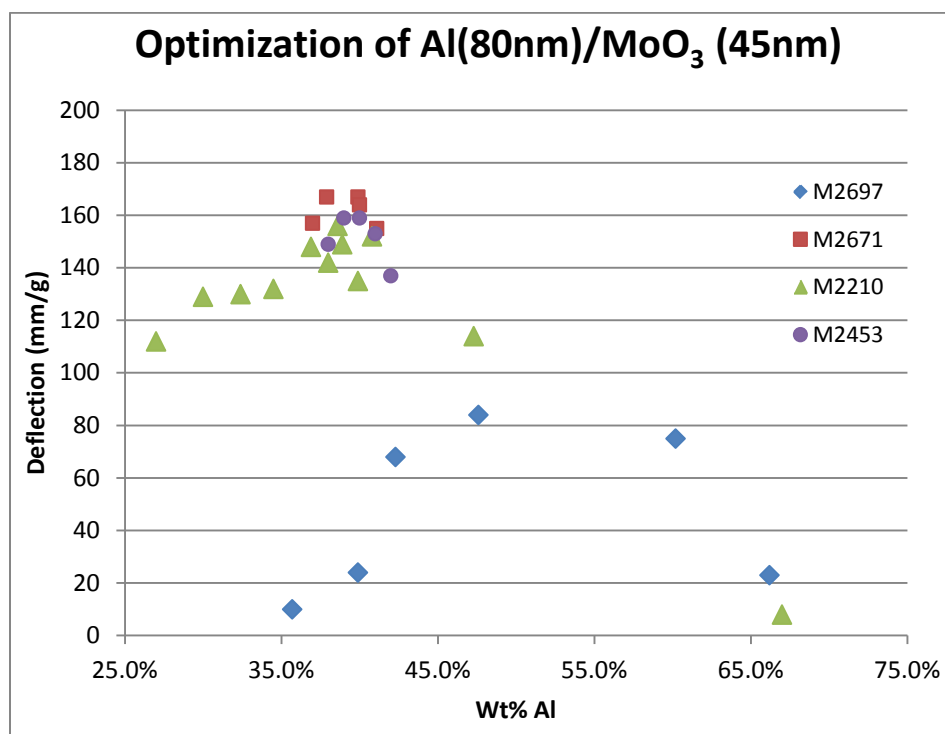
Figure 15: Optimization of several batches of 80 nm Al in the Al/MoO$_3$ ENC

A batch of 80 nm Al (M2697) was determined to be bad by the Al Pan Dent Test and it was not used to make LFEPs. The standard 40/60 Al/MoO$_3$ formulation gave a poor deflection number of 22 mm/g. Additional compositions were made using the M2697 batch that optimized at 50/50 but gave a poor deflection of only 82 mm/g. This batch of 80 nm Al powder was poor performing in all compositions. To determine the cause, the TGA of the M2697 batch of 80 nm Al was collected and exhibited poor oxidation behavior between 450-600°C as compared to the other batches. In this temperature range, the very small Al particles completely oxidize while the larger particles oxidize forming thicker passivation coatings and then stop oxidizing. M2697 has too high a ratio of large versus small particles that reduces it reactivity. The BET gave a larger

average particle size of 94 nm for M2697 than the 80 nm claimed by the vendor. China Lake notified and assisted NovaCentrix in identifying the problem. The growth conditions and parameters for M2671 and M2697 were identical except that a filter had been replaced between the 2 runs. The change in filter impacted gas flow rates and product particle size distribution. NovaCentrix and China Lake agreed that the problem could be avoided if the vendor pre-screens their material by TGA. Pure lead styphnate and lead azide were evaluated for comparison to the ENCs. The unconfined lead styphnate powder is ignited by the spark, flashed off without an audio report and gave a Deflection of 0 mm/g. The lead azide produced a sharp audio report and a large Deflection of 1080 mm/g. Lead azide is one of the highest performing materials examined to date by the Al Pan Dent Test and is exceeded by only the $Al(80nm)/AgIO_3(271nm)$ nanocomposite and copper azide (1088 mm/g).

6.1.1.4 Effect of Al Particle Size on Deflection - The choice of the optimal Al particle size is complex. As Al particle size decrease, the surface area increases, the active Al content decreases, the reactivity increases, and the amount of dead weight Al_2O_3 increases. Typically, 50 nm and 80 nm Al powders have active Al content of 62% and 74%, respectively. The advantage of 80 nm Al is higher active Al content while 50 nm Al has more reactive small particles. $Al/MoO_3(45nm)$ ENCs of 50 and 80 nm Al were optimized as shown in Figure 16:
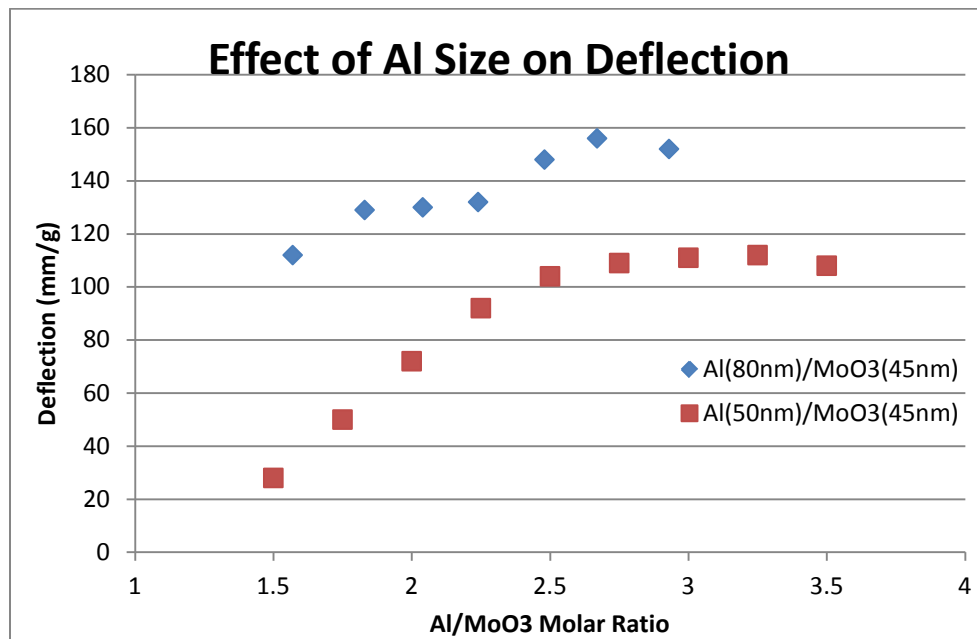
Figure 16: Al Size Effect on Deflection

The above figure shows that the 80 nm Al composites produce higher Deflection values (higher peak pressures) than the 50 nm Al composites. This is in part due to the nanocomposite energy content. The theoretical energy content for the Al/MoO_3 composite is -4.70 kJ/g. However, this energy is lower for nanocomposites because of the alumina passivation layer on the nano

aluminum powders. For 80 and 50 nm Al, the energy contents are -4.29 kJ/g and -4.03 kJ/g, respectively, when you account for the alumina dead weight.

6.1.1.5 Al Powder Summary – The 80 nm Al from NovaCentrix has been used to make high quality primers but monitoring of lot-to-lot variances must be tracked by TGA and the Al Pan Dent Test. Each lot should be optimized by the Al Pan Dent Test before LFEP formulation for product quality assurance. For 80 nm Al, the Al/MoO$_3$ ENC optimized at a molar ratio of 2.65 to 1. The stoichiometric molar ratio is 2 to 1 so the optimized nanocomposites are fuel rich.

6.1.2 Oxidizer Powders – The development of the Al Pan Dent Test enabled a re-examination of a wide variety of nanocomposites developed by the previous SERDP program. These materials plus new ones were characterized by the Al Pan Dent Test to identify potentially superior alternatives to the Al(80nm)/MoO$_3$ ENC used in the LFEP. A complete list of Al Pan Dent Test Deflections for ENCs can be found in Appendix K. Climax's MoO$_3$ (45 nm) was the oxidizer down-selected in the ESTCP project because of its commercial availability and its prior use in the SERDP program. A LFEP formulation using the nanocomposite, Al(80nm)/AgIO$_3$, showed outstanding promise in the SERDP program and served as a backup formulation in the ESTCP program. A major issue with AgIO$_3$ was the lack of a commercial availability of the nanopowders. Under a Defense Threat Reduction Agency (DTRA) project (MIPR # 10-2954M) commercial AgIO$_3$ micron powders were ball milled to suitable submicron size particle sizes and processed into ENCs (described in Appendix F). The DTRA project focused on the development of energetic agent defeat materials such as micron composites of Al/AgIO$_3$. The AgIO$_3$ milling process produced AgIO$_3$ that reacted rapidly with both micron and nano Al powders. The Al(80nm)/AgIO$_3$ ENC gave an outstanding Al Pan Dent Deflection of 953 mm/g.

6.1.3 Gas Additives – In order to attain AUR-ATs of less than 3.5 ms, a gas additive is needed to force the hot burning particles into the propellant bed. Primers without gas additives gave ambient temperature AUR-ATs between 40 to 60 ms. Although BTATZ worked well in the SERDP formulation, an alternative was needed since BTATZ was neither commercially available nor affordable. The properties of the ideal gas additive includes a large amount of gas release per gram (> 0.5 L/g), an exothermic decomposition, commercially available, cheap, and has a good thermal stability. Several gas additives were characterized using the Al Pan Dent Test including AN, BTATZ, DABT, Ammonium Perchlorate (AP), I$_2$O$_5$, and I$_2$O$_6$ to determine which parameters are critical.   BTATZ was tested as a baseline material since it had been used successfully in the SERDP program. The gas additives were added to the Al(80nm)/MoO$_3$ ENC in various quantities and the Deflection measured. This method helped to determine the optimal gas additive loading, a method to compare the performance of the additives, and a metric for down-selection.

The thermal decomposition of the gas additives are described in equations 4-8:

$$NH_4NO_3 \rightarrow N_2 + 2\ H_2O\ + \tfrac{1}{2}\ O_2 \qquad\qquad \Delta H = \text{-1.47 kJ/g} \qquad\qquad (4)$$

BTATZ ($C_4H_4N_{10}$) $\rightarrow$ 4/x(HCN)x + 3 N_2 (5)

DABT ($C_2H_4N_{10}$) $\rightarrow$ 2/x(HCN)x + 4 N_2 + H_2 (6)

ADN ($H_4N_4O_4$) $\rightarrow$ 2 N_2 + 2 H_2O + O_2 (7)

NH_4ClO_4 $\rightarrow$ ½ Cl_2 + ½ N_2 + 2 H_2O + O_2 ΔH = -1.60 kJ/g   (8)
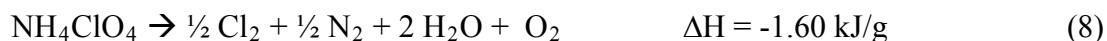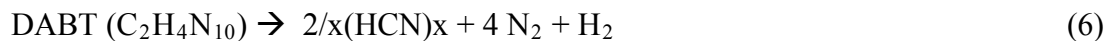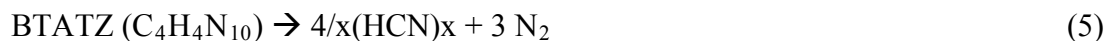
These gas additives were selected due to the large amount of gas generated per gram of material. The amount of gas released for AN, BTATZ, DABT, ADN, and AP are 1.38, 0.69, 0.58, 0.99, 0.89 L/g. I_2O_5 and I_2O_6 were also evaluated mainly because they produced oxidizing gases (I_2 and O_2). The oxididzing gases are reactive and should oxidize organic additives. The AN stands out as the top candidate for gas production per gram however, gas production has to be balanced with heat output. The maxmum loading for each gas additive is expected to be different and could be related to the amount of energy released during thermal decomposition.

6.1.3.1 Ammonium Nitrate - The effect of AN loading in the range of 0 to 60 weight percent is shown in Figure 17:
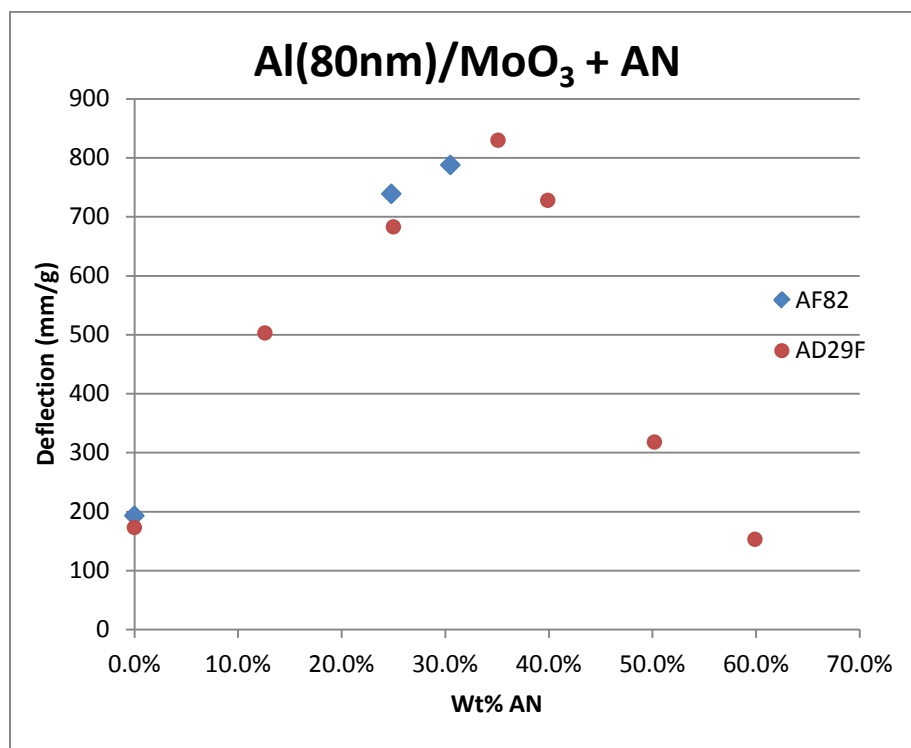
Figure 17: Effect of AN as a Gas Additive

The Deflection in Figure 17 increases to a maximum of over 800 mm/g at 35 wt% AN and then falls rapidly. The optimal range of AN additive was determined to be between 25 and 40%. AN was added to Al/MoO$_3$ ENCs, AF82 and AD29F, that utilized batches M2671and M2210 of 80 nm Al, respectively. The two Al batches gave similar deflections for the different AN loadings

and shows good batch to batch consisty even though the active aluminum content for the two batches were quite different (81.7% and 74.6%). LFEP samples with 10%(Y87), 20% (Y50), 30% (Y64) and 38 %(Y62) of AN, gave AUR-ATs of 3.70 $\pm$ 0.23ms, 3.07 $\pm$ 0.04ms, 3.04 $\pm$ 0.07ms, and 3.05 $\pm$ 0.07ms and LT AUR-ATs of 3.14 $\pm$ 0.09ms and 3.12 $\pm$ 0.09ms for Y64 and Y62, respectively. AN loading between 30 and 38% gave excellent AUR-ATs at ambient and at low temperatures. The AUR-ATs at ambient temperature are shown in Figure 18:
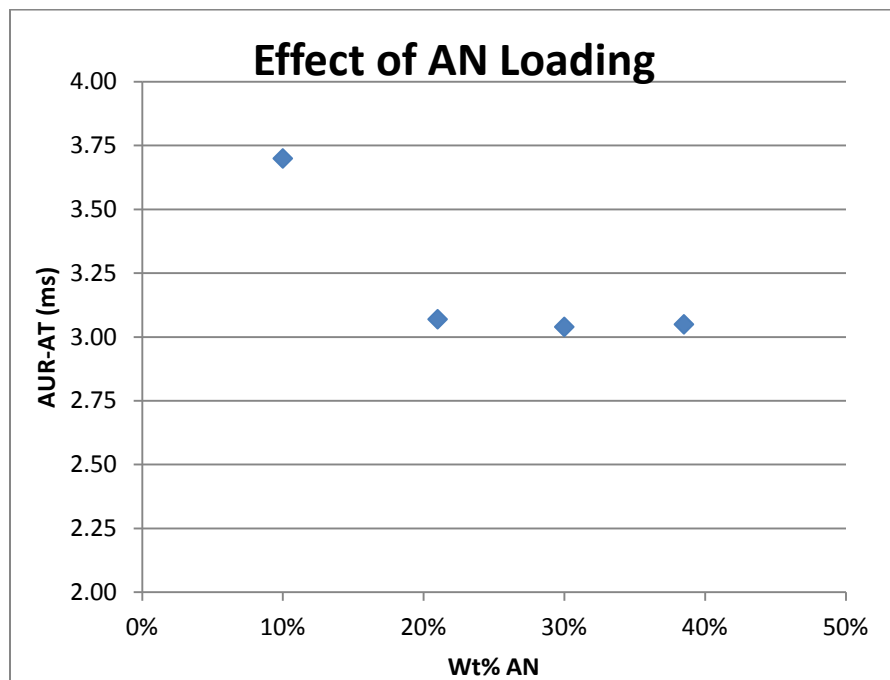
Figure 18: Effect of the Amount of AN Loading on AUR-ATs

A clear optimum AN loading was not apparent but excellent results were obtained even at a low AN loading level of 20% at ambient temperatures. A high AN loading is not necessarily desirable because of AN's low density (1.74 g/cc). The LFEP density decreases with increasing AN loading making it more difficult to consolidate 160 mg into a primer cup. Low LFEP charges (< 160 mg) resulted in longer AUR-ATs. In comparison, the standard load for a conventional M52A3B1 lead primer for 20 mm rounds is 178 $\pm$ 20 mg. Ideally, the LFEP would use the same loading quantity but the lower LFEP composite density made it difficult to put 178 mg into the primer cup.

6.1.3.2 High Nitrogen Compounds – BTATZ, ADN and DABT were compared as gas additives for the LFEP formulations. Although, BTATZ was used as the gas additive in the SERDP LFEP formulation and produced suitable AUR-ATs at ambient and low temperatures, it was not optimized with the Al Pan Dent Test. A maximum Deflection of about 200 mm/g was observed for 10 wt% BTATZ for a Al(80nm)/MoO$_3$/BTATZ composite. Both DABT and ADN

gave higher deflection numbers (high peak pressures) and allowed higher loading levels than BTATZ however AN was even better (> 800 mm/g). The Deflections versus wt% high nitrogen gas additive are shown in Figure 19:
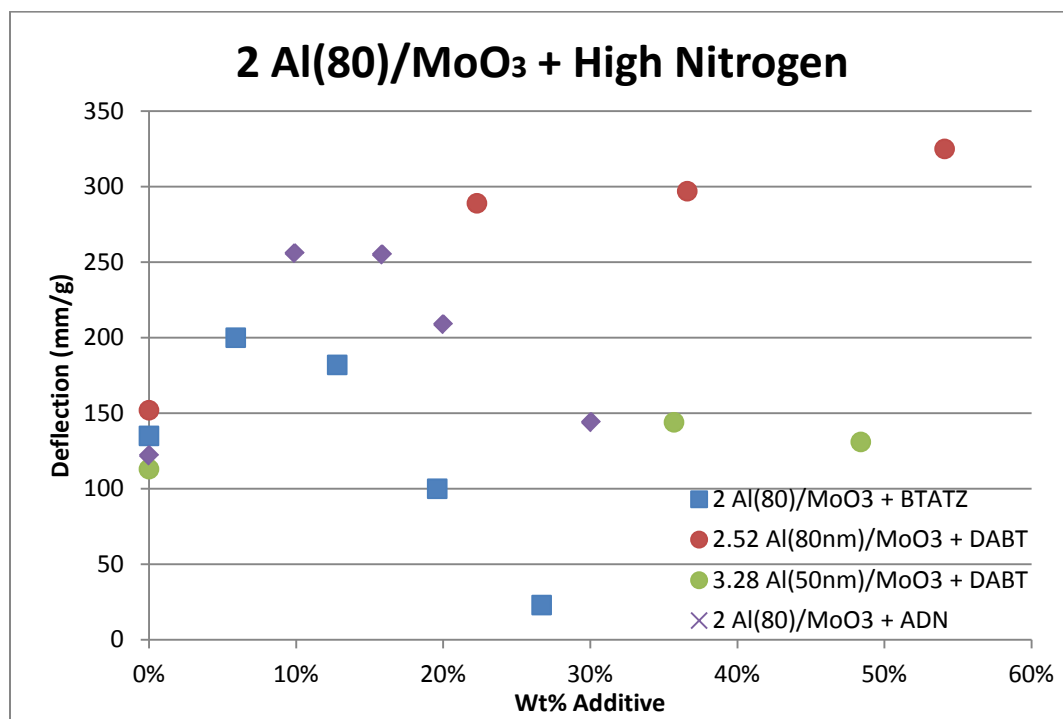
Figure 19: Al/MoO$_3$ + High Nitrogen Gas Additive

6.1.3.3 Ammonium Perchlorate (AP) - AP (17 microns) was examined as a gas additives because it is used in a wide variety of DOD applications. It is both a good gas generator producing nitrogen, water, oxygen and HCl and AP is a good oxidizer. AP gave excellent results as a gas additive and the Deflections for different weight percent AP are shown in Figure 20:

2.63 Al(80nm)/MoO$_3$ + AP
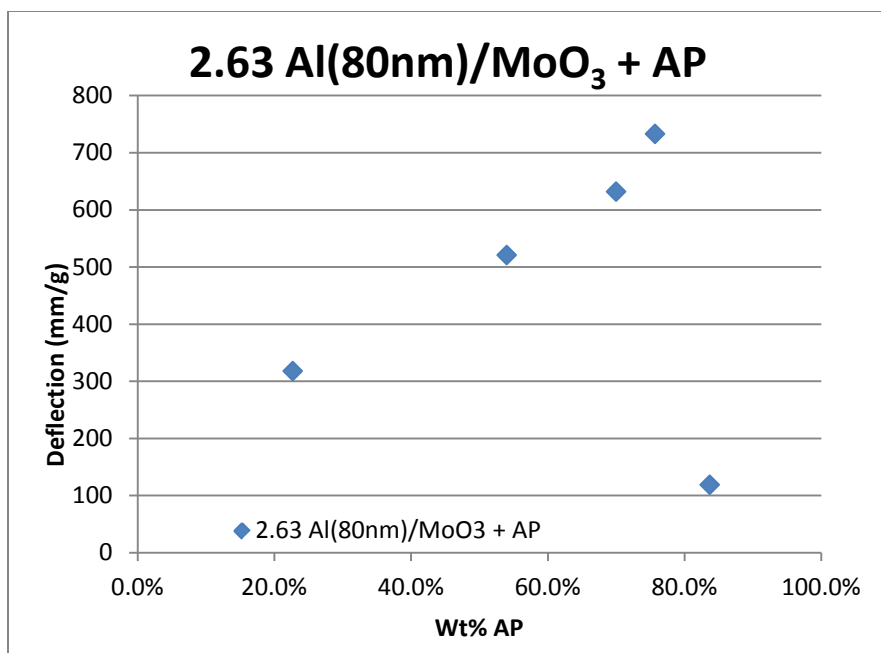
Figure 20: Ammonium Perchlorate as a Gas Additive

AP was the second best gas additives tested that also had the highest loading limit of 80 weight percent. Based on this data, a LFEP consisting of 19% Al(80nm)/MoO$_3$, 77% AP, 2% Kel-F and 2% Carbon could function as a medium caliber primer. However, as is the case for AN, the increased AP loading also decreases the LFEP density. A practical AP load limit appears to be around 40 - 60 weight percent. The ability to use a higher loading of cheap gas additives would lower the cost of the LFEP. Unfortunately, environmental concerns about AP contamination is currently a major concern.

6.1.3.4 Diiodine Hexaoxide – I$_2$O$_6$ is an oxidizer under development by a DTRA program. Like AP, it is potentially a very good gas additive. Thermal decomposition of I$_2$O$_6$ produces I$_2$ and O$_2$ via iodine oxide intermediates. The Deflections of Al/MoO$_3$ with I$_2$O$_3$ additive are shown in Figure 21:
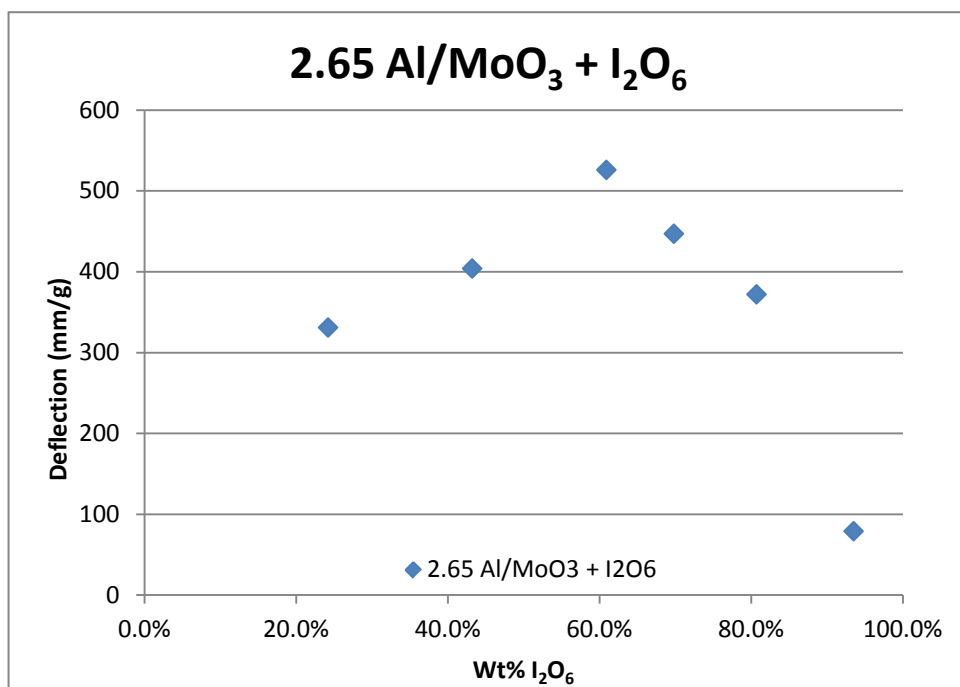
Figure 21: I_2O_6 as a Gas Additive

I_2O_6 gave the 3rd best result as a gas additive and represents a different class of additive in that the major gas generated is oxygen. The additional oxygen could increase the combustion rate and reduce the ignition delay of the nitrocellulose propellant.

6.1.3.5 Gas Additive Down-Selection - AN was selected because of the high commercial availability, low cost and satisfactory results in PVAT testing. The low density, phase stabilized AN obtained from Dyno Nobel is used as fertilizer and used to make the mining explosive, Ammonium Nitrate/Fuel Oil. It is abundantly available in pounds to train cart quantities, the manufacturer is stable, and the cost is very low (< $1/lb). The AN was characterized by BET, SEM and by formulating composites and evaluating the composites with the Al Pan Dent Test. In order to utilize AN as a gas additive, the material had to be milled. Ball milling of high density AN from Sigma-Aldrich led to very little particle size reduction. In contrast, low density AN in the form of prills from Dyno Nobel could be ball milled into suitable particle sizes. The ball milling of AN is described in Appendix G. Based on the gas additive testing results, any of the gas additives tested could function well in LFEP formulations. The ammonium nitrate phase change that is accompanied by a 3% volume change has been considered. This does not appear to be an issue with the primer because the LFEP composite is only 30% AN, the primer is consolidated to only about 72% of Theoretical Maximum Density and the LFEP is held together by the binder. The AN phase change represents less than 1% overall change in volume in a composite with 28% void space. In addition, 20 mm rounds fired after 3 & 6 months of storage in outdoor ammunition bunkers (50 to 115 °F) exhibited no detrimental effects.

6.1.4 Binder Additive – The binder plays an important role in the primer formulation. It maintains pellet integrity enabling the primer charge to withstand vibration and handling. LFEP without a binder suffered from poor reliability in the SERDP SEED effort. Kel-F binder, a DuPont product, was successfully used in the SERDP program and was used in the final SERDP formulation. However, at the start of the ESTCP program Kel-F was made only in Japan, requiring the replacement of Kel-F. Binders were evaluated in the ESTCP program (Teflon and other PCTFE) and compared to Kel-F. The binders were mixed into the Al/MoO$_3$ ENC in varying amounts and the Deflections measured with the Al Pan Dent Test. In the case of Teflon, it was observed that the maximum Teflon loading increased with increasing Al/MoO$_3$ molar ratio, 2.0 to 2.42 to 2.69, Figure 22:
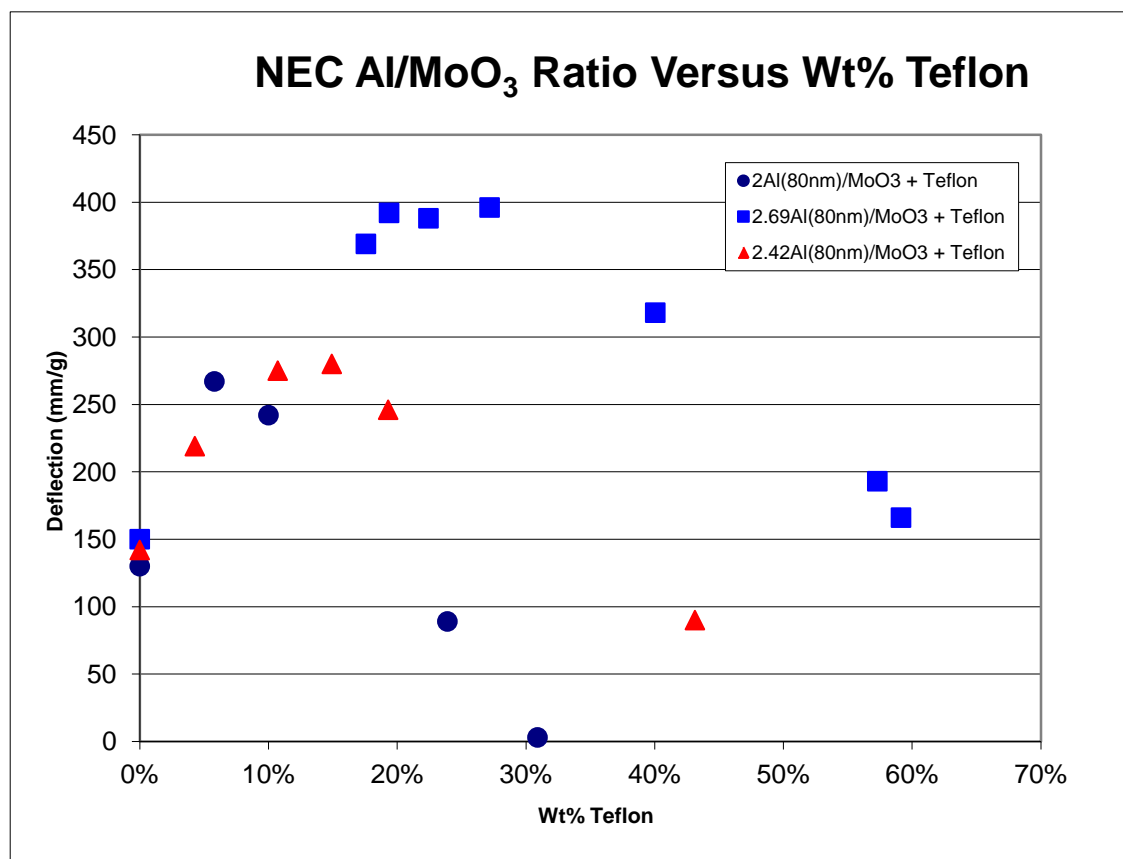
Figure 22: Effect of Teflon Addition for Different Al/MoO$_3$ Stoichiometries

In theory, fluorocarbon additives should react with the excess Al in the Al/MoO$_3$ composite leading to an increase in both energy and gas generation (equation 9):

$$4\ Al + 3/x\ (C_2F_4)_x \rightarrow 4\ AlF_3\ (g) + 6\ C \qquad \Delta H = -6.9\ kJ/g \qquad (9)$$

A comparison of the effects of Teflon and Halocarbon 600 (HC600) versus Kel-F loading is shown in Figure 23:
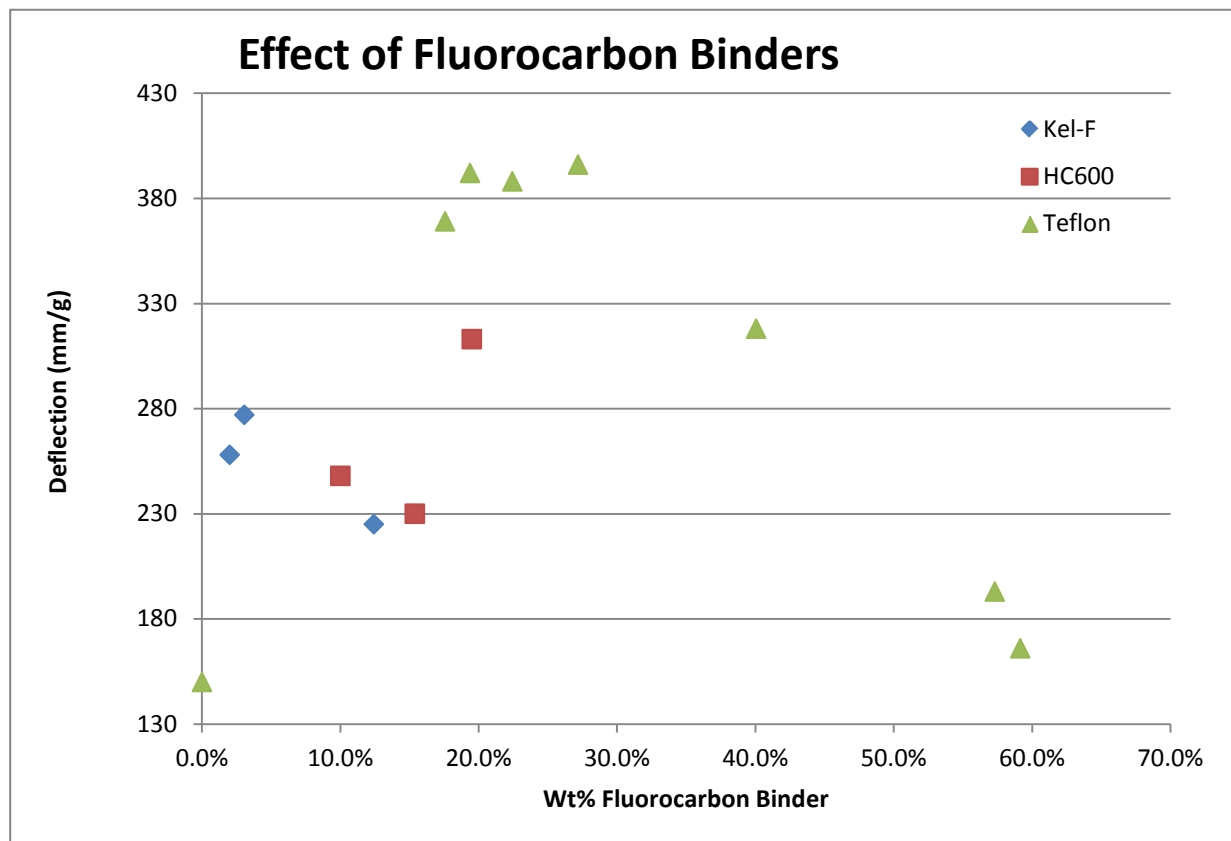
Figure 23: Effect of Different Fluorocarbon Binders on Deflection

Based on the Deflection data, both HC600 and Teflon can be used at much higher loading levels than Kel-F. Halocarbon 600 and Kel-F are polychlorotrifluoroethylene (PCTFE) polymers and the reaction with aluminum is exothermic, equation 10:

$$4 \text{ Al} + 3/x \text{ } (C_2ClF_3)_x \rightarrow 3 \text{ AlF}_3 \text{ (g)} + AlCl_3 + 6 \text{ C} \qquad \Delta H = -6.2 \text{ kJ/g} \qquad (10)$$
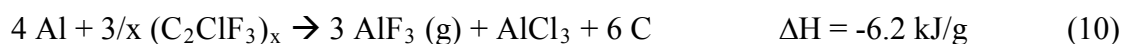
However, in high speed videos of nanocomposite and micron composite combustion, the burn rates decrease with increasing halocarbon content. The high carbon-fluorine bond strength appears to cause an ignition delay in LFEP as well. When Teflon was used to replace Kel-F in the standard KTHZ60 (AUR AT = 3.04 ms) formulation, 66% Al/MoO$_3$, 30% AN, 2% Kel-F, 2% C, the primers failed to fire. This was observed for KTHZ72, KTHZ10 and KTHAB4. In a formulation where Kel-F and Teflon was used in combination (2% Kel-F and 2% Teflon), an AUR-AT of 12.8 ms was observed at ambient temperature. Even when Teflon was added as a mixture it produced poor AUR-AT results. Although the addition of fluorocarbon binders increase the LFEP energy content, the quantities used must be kept low due to ignition delays. **Kel-F binder was down-selected for use in the ESTCP formulation since it is now made in**

many countries including the U.S. The fluorocarbon binder also served to decrease the ESD sensitivity making the ENC safer to handle. The desensitization of ENCs with fluorocarbon additives was patented.[24,25]

6.1.5 Conductive Additive – Chevron-Phillips' (CP) acetylene carbon black works well in the LFEP formulation but also adds limitations. LFEPs only fire if the carbon is added via dry mixing. In an attempt to add the carbon in hexane, the carbon was placed in hexane and then sonicated. The sonicated carbon/hexane slurry was quickly added to the Al(80nm)/MoO$_3$/AN/ Kel-F/hexane slurry and stirred for 1 minute. The hexane was removed and primers pressed dry. All attempts to fire AURs failed. Alternative conductive additives were investigated to replace the carbon black. Primers that replaced CP carbon with graphite (KTHAB4), zinc dust (KTHAB5), and Ag flake (KTHAD6) were investigated. The LFEP made using these alternative conductive additives all failed to fire even though the additives were mixed in dry. The failure of these alternative additives may be due to their large particle size. The large particles may have created a conductive pathway from the primer cup button to the walls (shorting the system) or there weren't enough particles to enable a spark to jump from particle to particle. **Only Acetylene Carbon Black gave acceptable results.**

6.1.6 Material Down-Selection Summary - The 80 nm Al from NovaCentrix, 45 nm MoO$_3$ from Climax, AN from DynoNobel, Kel-F from Halocarbon, and Acetylene Carbon Black from Chevron-Phillips were down-selected for the LFEP. Based on the Al Pan Dent Testing, the preliminary LFEP composition was 66% Al(80nm)/MoO$_3$(45nm) with a 2.65 $\pm$ 0.02 molar ratio, 30 $\pm$ 5% ammonium nitrate, 2 $\pm$ 0.2% Kel-F and 2 $\pm$ 0.2% Acetylene Carbon Black.

6.1.7 Summary of Characterizations for Starting Materials - The recommended characterization techniques for each of the LFEP components are listed in Table 5:

Table 5: Recommended Characterization for LFEP Materials

	TGA	Hydrolysis	BET	SEM	X-Ray	Fourier Transform-Infrared Spectroscopy	ESD	Al Pan Dent Test
Metals	Yes	Yes	Yes	Yes	Optional	No	Yes	Yes
Oxidizers	Yes	No	Yes	Yes	Yes	Optional	Yes	Yes
Gas Additive	Optional	No	Yes	Yes	Optional	Optional	Yes	Yes
Binders	Optional	No	Optional	Yes	Yes	Optional	No	Optional
Conductive	Yes	No	Yes	Yes	Optional	No	Yes	Optional

Additive								

6.2 MATERIAL FORMULATION AND OPTIMIZATION

The LFEP formulation process evaluated a variety of parameters including fuel-oxidizer ratio for the ENC, order of mixing, alternative components, alternative solvents, mixing processes, and scale-up effects on performance and safety properties. The LFEP formulation and optimization processes in the SERDP SEED, Full program and initially in the ESTCP program were completely Edisonian approaches of trial and error. LFEP compositions were formulated and tested by firing all-up rounds to obtain action times. Variables such as amount of gas additive were changed arbitrarily to see what effect it had on the AUR-ATs. This approach led to little understanding of the critical factors affecting the all-up round action times. The development of the Al Pan Dent Test enabled a wide range of material compositions to be evaluated before transitioning to the all-up round testing phase.

6.2.1 Small-Scale Formulation and Optimization

6.2.1.1 Effect of Al/MoO$_3$ Molar Ratio on AUR-ATs – The optimization of various batches of 80 nm Al with MoO$_3$(45nm) gave an optimum Al/MoO$_3$ molar ratio of 2.67. A series of LFEP with the composition of Al(80nm)/MoO$_3$ (66%), AN (30%), Kel-F (2%) and C (2%) were prepared where the Al/MoO$_3$ molar ratio was varied from 2.5 to 2.77 to determine if the molar ratio of the ENC impacted the AUR-AT. The impact of varying the fuel-oxidizer ratio of the ENC is shown in Figure 24:

Effect of Al/MoO$_3$ Molar Ratio on AUR-ATs
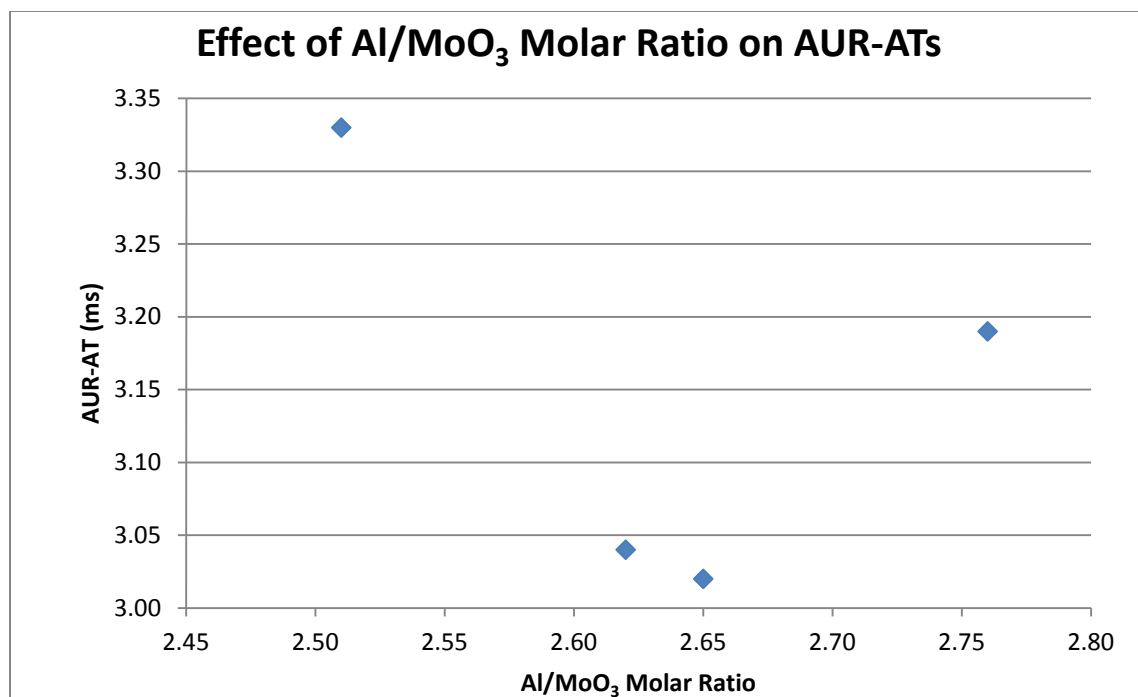
Figure 24: Effect of Al/MoO$_3$ Ratio on AUR-ATs

The AUR-ATs reaches a minimum when Al/MoO$_3$ molar ratio is around 2.65 which was consistent with the optimal ratio of 2.67 found for the Al(80nm)/MoO$_3$(45nm) ENC. **The optimization of the ENC is a critical parameter for LFEP performance.**

6.2.1.2 Standard Final LFEP Composition - The final LFEP composition was identical to the preliminary proposed composition based on laboratory testing. The Standard LFEP formulation is based on KTHZ60 with the composition, 66% Al(80nm)/MoO$_3$(45nm) with a 2.66 $\pm$ 0.03 molar ratio, 30 $\pm$ 2% ammonium nitrate, 2 $\pm$ 0.2% Kel-F and 2 $\pm$ 0.2% Acetylene Carbon Black. Formulations consistent with the standard LFEP formulation will be denoted as **Standard LFEP (KTH***)** with the batch name in parenthesis. The **Standard LFEP** composition was chosen due to the best AUR-ATs in the PVAT testing.

6.2.1.3 LFEP Alternate Mixing Sequence – The **Standard LFEP** was prepared but the mixing sequence changed to examine if coating the Al/MoO$_3$ ENC with Kel-F first would improve the LFEP. This approach would place nearly all of the Kel-F binder on the ENC and should reduce the ENC's ESD, Impact and Shock Sensitivity. The Al(80nm)/MoO$_3$/Kel-F was then mixed with AN and then dry mixed with carbon. The AUR-AT of Standard LFEP (KTHY84) prepared by the alternate process was slightly higher at 3.19 $\pm$ 0.13 ms. The slightly higher AUR-ATs are consistent with high speed imagery that showed longer slower burn times when ENCs are coated with binders. **The normal mixing sequence was down-selected.**

6.2.1.4 Finalized Small-Scale (1g) Lead-Free Electric Primer Process

The Al/MoO$_3$ (0.66g) ENC is placed in a 50 ml polyethylene container, 25 ml of hexane added, AN (0.30 g) added, stirred for 1 minute, allowed to settle for 2 minutes and then the excess hexane removed with a pipette. Kel-F (0.02g) is dissolved in 5 ml of hot hexane and added to the Al/MoO$_3$/AN slurry. The slurry is stirred for 2 minutes and then the hexane evaporated off under a mild stream of air in a hood. The product is placed on a grounded Al foil and the chunks broken down with a grounded spatula. The product is transferred to a grounded metal can (2.5" diameter by 2" height), carbon black (0.02g) added, the can closed and mixed by agitation for 1 minute.

6.2.1.5 Finalized LFEP Mixing Process (5g, 10g, 25g) – Al/MoO$_3$ (3.30g) was prepared in 1.1g batches and transferred into a 150 ml polyethylene cup. The AN (1.50g) was added and stirred for 1 minute, allowed to settle for 2 minutes and the excess hexane removed. Kel-F (0.10g) dissolved in 25 ml of hot hexane was added to the Al/MoO$_3$/AN slurry, stirred for 2 minutes, and then the hexane evaporated off under a mild stream of air in the hood to a paste (not dry). About 1g of material is transferred to a sheet of Al foil, air dried and broken down to a fine powder. This is repeated 4 more times, keeping the dry product separated. The powders are added to a grounded metal can one at a time, the carbon black added (0.10g) and dry mixed using a roller behind a shield for 2 minutes (about 30 rpm). Hexane (25ml) is added to the LFEP material and transferred into conductive vials. This same procedure was used for 10g and 25g batches by combining 5g batches in hexane.

The Al Pan Dent Test Deflections for the precursors and LFEP composites used to optimize the LFEP can be found in Appendix M.

6.3   LFEP SCALE-UP PROCESSES

The use of an ultrasonic horn to mix/activate the ENC is not a practical process for production since the maximum sample size is limited to 2g per batch. In order to make 10,000-100,000 primers per day, a process that can produce 1.6 to 16 Kg of Al/MoO$_3$ nanocomposite per day is needed. A bulk method was needed to make ENCs and some potential issues with scale-up are:

1) Performance – Will the scaled-up material perform as well as the small scale?
2) Will the scale-up process impact the hazard properties?

6.3.1 ENC Scale-Up Methods

6.3.1.1 Resonance Acoustic Mixer (RAM) – The Resodyn RAM mixing system was evaluated to determine if this new technology could produce high performance ENCs in bulk. Six samples were prepared and mixed using the RAM technology. Three 1g samples of Al(80nm)/MoO$_3$ in 25 ml of hexane were mixed at 47 "g" for 30 , 20 and 10 seconds. Two 1g samples of Al(80nm)/AgIO$_3$(City) and a ALEX/AgIO$_3$ were mixed in hexane at 50"g" for 20 seconds. A 5g sample of Mg/Teflon was mixed in hexane at 100"g" for 20 seconds using the RAM. Isolation and characterization of the test samples all gave poor Al Pan Dent Test results. The fuel and

oxidizers settled out separately and even the demonstration samples brought by the Resodyn vendor separated on standing. **The RAM system was a total failure for the bulk preparation of energetic nanocomposites.**

6.3.1.2 NAWCWD's Modified Ball Milling Scale-Up Method – Ball milling of micron thermite materials is not new. Many laboratories have published data especially Professor Edward Dreizin at NJIT.[26] However, when micron starting materials are used, there is a critical point where the starting materials react during the milling process. To avoid the reaction during milling, NAWCWD modified the standard method by utilizing a soft housing (Polyethylene Bottles) and using nanopowder ingredients. This represents a low-energy milling process essential for large scale ENC preparation. Two types of milling balls have been evaluated (Alumina and Stainless Steel). A scale-up patent for the preparation of energetic nanocomposites has been issued by the US Patent and Trademark Office.[27]

6.3.1.2.1 Al/MoO$_3$(45nm) ENC Composite Preparation By Modified Ball Milling – An example procedure of a modified ball milling preparation is described for Al(50nm)/MoO$_3$(45nm) KTHZ2. A 250 ml polyethylene (PE) bottle was loaded with 5.86g of 50 nm Al powder, 6.10g of MoO$_3$(45nm) powder, 50ml of hexane and 10 alumina balls (26g). The bottle was wrapped with electrical tape to increase friction between the roller and the bottle. The bottle was placed on a US Stoneware Roller set at 50% power. Samples of the product were removed periodically and tested using the Al Pan Dent Test. The mixing was stopped once the deflection stabilized. The Deflection versus milling time for several different Al/MoO$_3$ composites is shown in Figure 25:
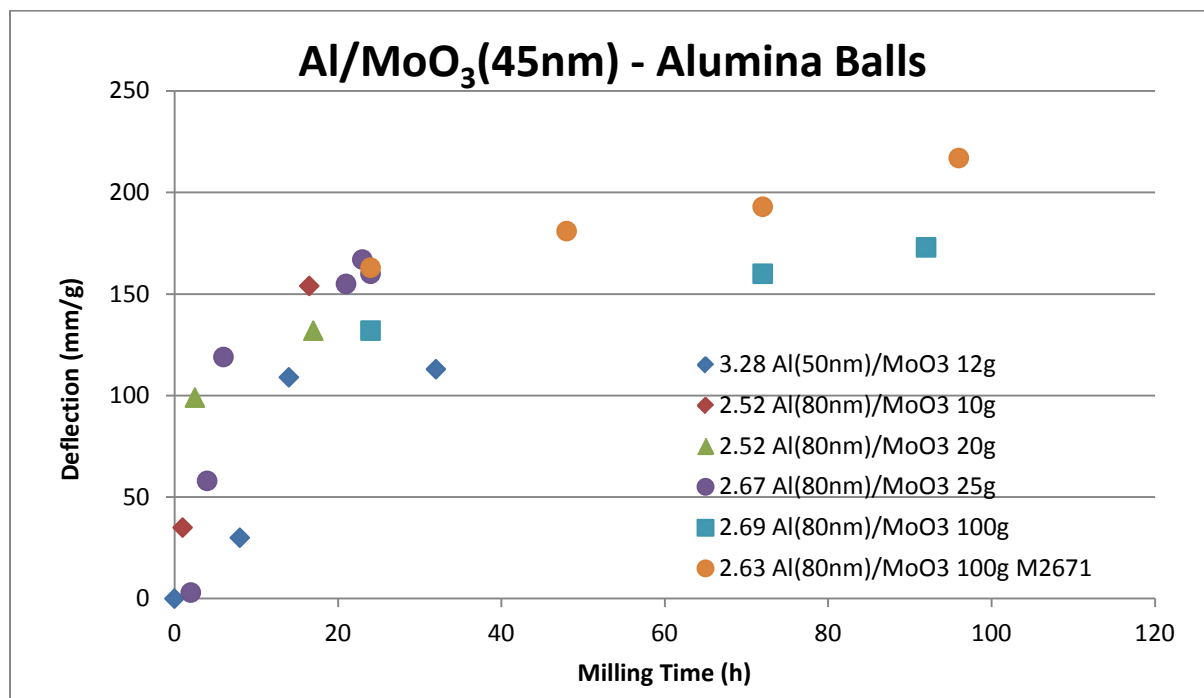
Figure 25: Modified Ball Milling Using Alumina Ball Milling Media

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The milling time to reach maximum performance is unusually long requiring several days. However, most composites prepared by the modified ball milling process were as good as or better than identical samples prepared by small scale sonication methods. The best Al/MoO$_3$ ENC using 80 nm Al gave a Deflection of 160 mm/g. The ball milled samples often exceeded 220 mm/g which is indicative of better fuel-oxidizer mixing. A safety issue was found relating to the use of ceramic milling balls. During the milling process, the milling balls pick up charge via tribo-electric charging. In one case, a grounded spatula ignited a ceramic milling ball causing a small fire.

6.3.1.2.2 Al/MoO$_3$(45nm) ENC Composite Preparation By Modified Ball Milling With Steel - The effect of changing the milling media from alumina to stainless steel was investigated. The alumina balls are porous with a diameter of 12.5 mm and density of 2.21 g/cc as compared to the stainless steel balls with a diameter of 5 mm and density of 7.86 g/cc. The three samples all used the same starting materials, 80 nm Al (lot M2671) and MoO$_3$(45nm). The compositions were kept nearly constant but the Ball to Product Ratio (BPR) varied along with the amount of material. Figure 26 shows the Deflection versus milling time comparing stainless steel versus alumina milling balls:
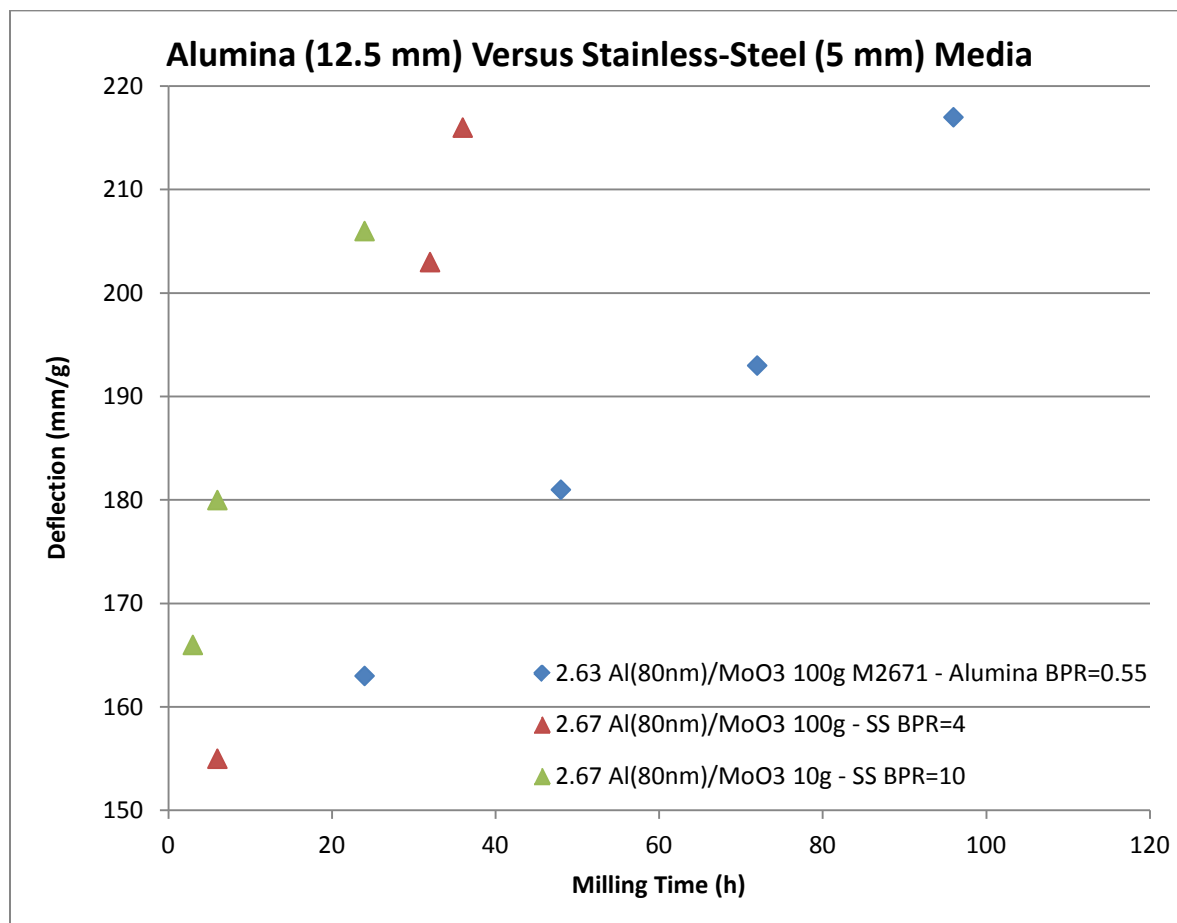
Figure 26: Alumina Versus Stainless Steel Media

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The higher density of the steel balls also enabled higher BPRs than for alumina. Due to the low density of the alumina balls the 100g run had a BPR of 0.55 while the batch using the steel milling balls had a BPR of 4. The alumina batch required 4 days to exceed 200 mm/g deflection while the stainless steel batch reached over 200 mm/g is less than 36 hours. For a smaller 10g run using stainless steel and a higher BPR of 10 exhibited an even faster rise in deflection versus time. The faster rise is due to smaller size and higher BPR. The use of stainless steel milling balls enabled reduced milling time because of the higher density of steel (7.86 g/cc) versus porous alumina (2.21 g/cc).

Additional modified ball milling runs for ALEX/MoO_3, Al(80nm)/Bi_2O_3, and Al(80nm)/$AgIO_3$ can be found in Appendix J. High performance materials were generated by the modified ball milling process. All scale-up runs to produce Al(80nm)/Bi_2O_3 and Al(80nm)/$AgIO_3$ ENCs were funded by DTRA. The scale-up runs of other nanocomposites validated the usefulness of the modified ball milling scale-up approach. The Al Pan Dent Test deflection for almost every ENC prepared by NAWCWD's Modified Ball Milling process is equal or better than the best result obtained by the conventional small scale sonication process. In addition, LANL compared Al(80nm)/MoO_3 prepared by sonication and modifed ball mill in their pressure cell and found that the ball milled product gave a slightly higher peak pressure and higher impulse.

NAWCWD's Ball Milling process has been scaled up from 10g to 100g with no difficulty. Further scaling to many kilograms is possible but should be done by the industry. The ENC product from the bulk milling process is also superior to the small scale sonication method and can be used to make a variety of different ENCs. A comparison of the Deflection number of sonicated samples and ball milled samples is shown in Table 6 and additional Deflections for additional scale-up samples are listed in Appendix K:

Table 6: ENCs Prepared by Sonication and Modified Ball Miling

Name	Composite	Al/Oxidizer Ratio	Deflection (mm/g)	Method
Y46F	Al(80nm)/MoO_3(45nm)	2.67	154	Sonic
AF14	Al(80nm)/MoO_3(45nm)	2.67	217	BM
AD32	Al(80nm)/$AgIO_3$(271 nm)	2.14	1095	Sonic
AE80	Al(80nm)/$AgIO_3$(277 nm)	2.19	1308	BM

The Al(80nm)/$AgIO_3$ ENC was developed as the backup material for Al(80nm)/MoO_3 in the LFEP in the SERDP program. However, DTRA (MIPR # 10-2954M) funded further development of this ENC. The preparation of various $AgIO_3$ particle sizes, the purchasing of starting materials, the ball milling of commercial $AgIO_3$ and the preparation of large scale batches of the Al(80nm)/$AgIO_3$ were funded by DTRA not the ESTCP. The Al(80nm)/$AgIO_3$(277nm) ENC made by ball milling (1308 mm/g) is by far the best performing material tested to date. The most likely reason for this is that the sonication process does not completely break up Al agglomerates in hexane and that re-agglomeration also occurs. The ball

milling process appears to be more efficient at breaking down and mixing the agglomerated fuel with the oxidizer. Examination of sonicated and ball milled samples of Al(80nm)/MoO$_3$ appear to be identical by SEM. The SEMs can be found in Appendix L.

6.3.1.2.3 Al(80nm)/AgIO$_3$/AN/Kel-F/C Backup LFEP Formulation - As a risk reduction effort in the SERDP program, a high performing ENC was used in place of the standard Al(80nm)/MoO$_3$ ENC (148 mm/g) in the LFEP formulation. The Al(80nm)/AgIO$_3$ ENC was optimized using the Al Pan Dent Test and gave a deflection of 973 mm/g for a composite with a 2.04 Al/AgIO$_3$ molar ratio. A LFEP composite (KTHY82) was made containing 71 wt% Al(80nm)/AgIO$_3$(235nm), 25 wt% AN, 2 wt% Kel-F and 2 wt% carbon, loaded and fired on the Mann Barrel gun at ambient and low-temperature. This produced the best AUR-ATs to date for any LFEP, 2.92 $\pm$ 0.08 ms at ambient and 3.01 $\pm$ 0.02 ms at -66 °F. Although no ESTCP work was performed on this backup formulation, progress was made off of DTRA funding (MIPR #10-2954M). Al(80nm)/AgIO$_3$ ENCs were made and characterized. Micron AgIO$_3$ powder is commercially available and ball milling produced suitable particle sizes for LFEP application.

6.3.2 Scale-Up of Standard LFEP – The LFEP formulation went through a number of iterations to removal hazardous steps and to minimize the number of steps in the formulation while not affecting the LFEP performance.

6.3.2.1 Preparation of 100g of LFEP - A 1L PE bottle is charged with 400g of 5 mm stainless steel milling balls, 28.35g of 80 nm Al, 41.65g of 45 nm MoO$_3$, and 700 ml of hexane. The bottle is closed and placed on a US Stoneware Roller set at 100% power in a hood behind shields. The material is milled for 36 to 48 hours. A sample of the Al/MoO$_3$ is taken out for Al Pan Dent Testing and the deflection number should be at least 190 mm/g. The low density AN (30.0g) is added to the PE bottle and rolled for 30 minutes. The product is then isolated from the steel milling balls by decanting the product into a 2nd 1L PE bottle through a grounded metal screen. This is done in a hood with a 2L grounded Al tray sitting on a large grounded tray to capture any spillage. The product will settle to the bottom of the PE bottle and the hexane is decanted off the product into the original milling bottle. The milling bottle is swirled to separate off product from the steel milling balls and the slurry decanted into the 1L PE bottle. This step is repeated twice more to capture as much of the product as possible (A total of 4 decanting, 3 of which are washings). Water (500 ml) is added to the milling bottle containing the steel balls and some residue. This will deactivate the Al/MoO$_3$/AN composite within 30 minutes. Excess hexane is removed from the settled product by decanting. A hot solution of Kel-F (2g) dissolved in 50 ml of hexane is added to the product. The bottle is closed and placed on the roller for 15 minutes where the Kel-F slowly precipitates out of solution during the mixing-cooling period. The carbon addition step was performed dry. Several attempts to add the carbon in hexane resulted in no-fires. Carbon was added to hexane, added to the Al/MoO$_3$/AN/hexane slurry and stirred for 30 minutes (KTHAB95). In another experiment, the carbon black was dispersed in hexane using a 400 W ultrasonic horn then added to the Al/MoO$_3$/AN/Kel-F/hexane slurry, and

stirred for 30 minutes. The carbon black was also coated with Palmitic acid in order to increase dispersion then added. In all cases, No-fires resulted. Successful firings have only occurred when the carbon was added as a dry powder to dry Al/MoO$_3$/AN/Kel-F powders.

6.3.2.1.1 Scale-Up of Carbon Addition – To mitigate risk to workers during the carbon addition step of the LFEP preparation, a 100g carbon mixer was designed and fabricated. The mixer is shown below in Figure 27:
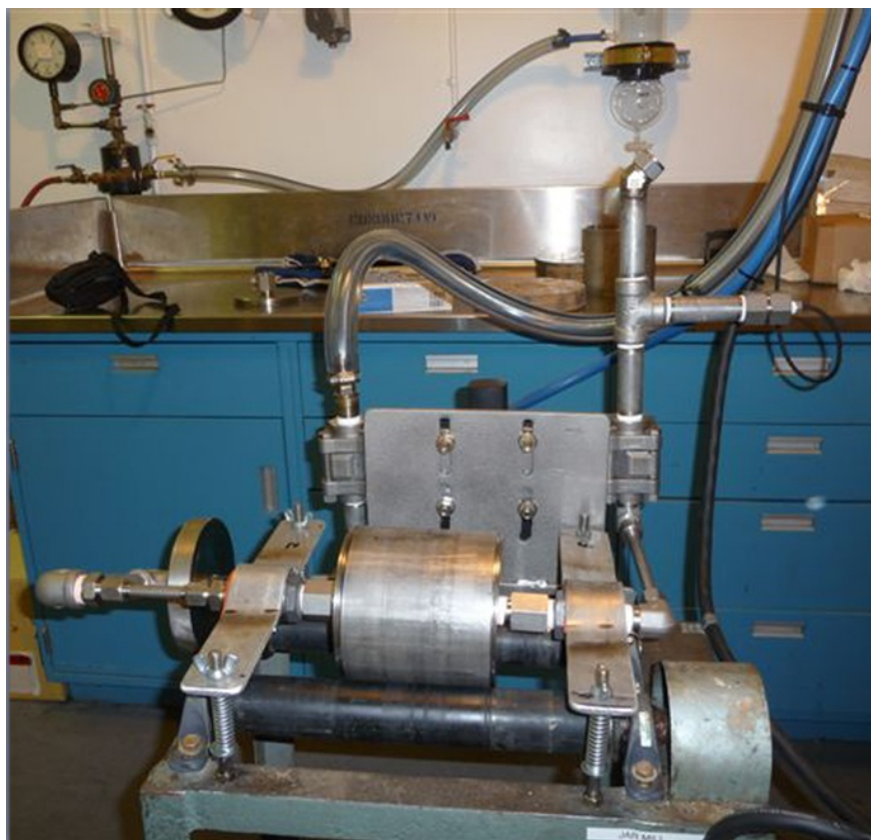
Figure 27: Stainless Steel Dry Mixer

The 100 g mixing system is made up of a grounded stainless steel mixing chamber, the air actuated valves, a solvent trap system, and a roller that is driven by an explosion proof motor. The mixing chamber is attached on both ends with rotating joints with filters attached to prevent ENC from contaminating the trap system. The mixing chamber has removable top and bottom covers to enable easy access to the primer material.

Mixing Procedure: The mixing chamber is loaded with the Al/MoO$_3$/AN/Kel-F slurry in hexane. The chamber is attached to the vacuum/solvent trapping system on the rollers. The chamber is rotated at a rate of about 30 rpm and the hexane removed under vacuum and collected in a -78°C trap. Carbon powder is loaded in one of the side arms and a solenoid valve is opened allowing the carbon to be transported into the mixing chamber. The mixing chamber is re-

pressurized to atmospheric pressure and the carbon is mixed by rotation of the mixing chamber for 30 minutes.

PVAT Testing – The AUR-ATs using material with the Standard LFEP composition gave unusually high numbers, > 40 ms. Laboratory testing showed that the carbon mixing was very heterogeneous with almost no carbon to high levels of carbon. **This mixing method was abandoned.**

6.4   20 MM ALL-UP ROUND TESTING, LOADING AND CONSOLIDATION OF LFEP

A number of parameters during the loading and consolidation process of the LFEP influe the ambient and low temperature AUR-ATs, data scatter, and overall performance. A number of parameters were investigated including primer load weight, consolidation pressure, wet versus dry loading, die configuration, consolidation of small versus scaled-up LFEP, and consolidation using the multi-die set.

6.4.1 LFEP Loading Weight – For the M52A3B1 primers, a critical primer load weight of 178 $\pm$ 20 mg is specified. In order to determine the optimum amount and minimum amount of LFEP needed to produce suitable AUR-ATs, loads of 145, 150, 155 and 160 mg were loaded into primer cups and fired in the Mann barrel. Figure 28 shows the effect of LFEP load weight on the AUR-AT at ambient temperature. The LFEP samples were prepared with the same composition but one utilized Al(80nm)/MoO$_3$ made by sonication (AD54) and ball mill (AD60).
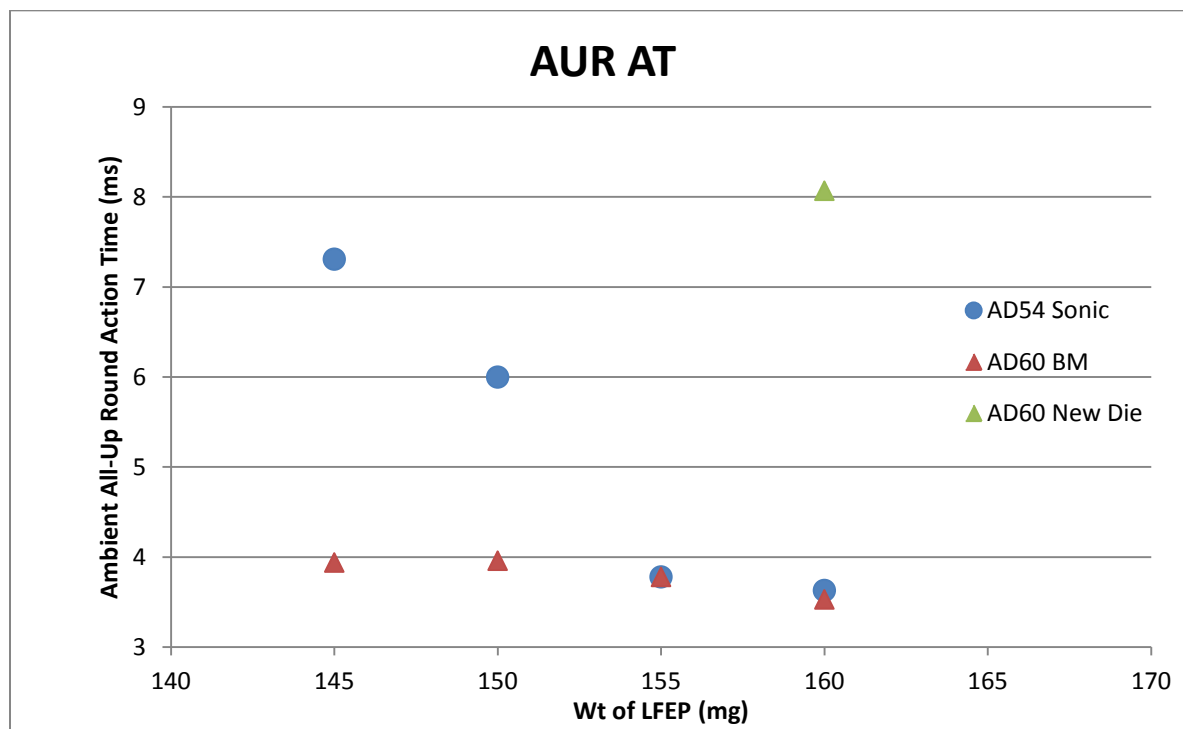
Figure 28: Effect of LFEP Loading Weight On AUR-ATs

The standard LFEP primer composition utilizing the Al/MoO_3 ENC made by sonication exhibited a significant increase in AUR-ATs at low LFEP loading levels (< 155 mg). At 145 and 150 mg, unacceptable AUR-ATs of 6 to 8 ms were obtained. In contrast, the LFEP based on the ball milled Al/MoO_3 ENC gave AUR-ATs below 4 ms. The graph suggests that the minimum amount of LFEP should be at least 165 mg or more.

6.4.2 Consolidation Pressures – The effects of consolidation press is a critical parameter especially for multi-die pressing. In any multi-die configuration, there will probably be variances in pressure between the center, edges and corners of the matrix. An understanding of the AUR-AT dependent on consolidation pressure was needed. To address this need, the LFEP standard formulation (KTHZ60) was consolidated at 6000, 6500, 7000, and 7500 psi, and fired in the Mann barrel. This resulted in AUR-ATs of 3.05 ± 0.08, 3.03 ± 0.08, 3.01 ± 0.05, and 3.02 ± 0.09 ms, respectively. The AUR-ATs for the 4 pressures are virtually identical and the average for all of the pressures was 3.02 ± 0.05 ms. The lack of sensitivity to variances in consolidation pressure makes potential of the multi-die set very promising.

6.4.3 Dry Versus Wet Loading – Both dry and wet (solvent) loading were investigated to increase production process options and to identify safer/useful processing conditions. The dry loading procedures were described in section 5. The advantages of a dry loading process include the use of a powder injector system for loading the primer material into primer cups, not having to add solvent to the primer powder, and fewer pressing steps. The disadvantage is dealing with a hazardous very ESD sensitive solid. Although the hazard can be managed with proper electrical grounding and use of personal protection equipment, human error has resulted in a number of mishaps nationwide with the related Al/Bi_2O_3 ENC. Only one incident has occurred with the Al/MoO_3 ENC where about 10g ignited in a humidified glovebox at General Dynamics Ordnance and Tactical Systems (GDOTS). The advantages of wet loading are the reduced hazard risk and the fact that LCAAP currently uses a water loading system for their current primer production. The Army and North Dakota School of Mines and Engineering are investigating the use of water preparation and loading of lead-free percussion primers. In this program, the effects of loading using different organic solvents were compared to dry loading. Table 7 is a summary of the AUR-ATs of dry versus wet loaded LFEP with the standard down-selected composition. The batch names are different but the compositions are consistent with the Standard LFEP composition.

Table 7: AUR-ATs of Different Batches of the Standard LFEP Loaded Wet and Dry

DRY Loaded				Wet Loaded				
Standard LFEP Batch	AUR-AT ms	STDEV	Drying Step	Standard LFEP Batch	AUR-AT ms	STDEV	LT AUR-AT	STDEV
KTHAF144	3.25	0.14	Yes	KTHAF144	3.21	0.14		
KTHAF80	3.98	0.42	No	KTHZ60	3.02	0.09		
KTHAD60	3.11	0.02	No	KTHZ60	3.01	0.05		
KTHAD60	3.21	0.11	No	KTHZ60	3.03	0.08		

KTHAD56	3.53	0.32	No	KTHY64	3.04	0.07	3.17	0.09
KTHAD54	3.59	0.23	No	KTHY62	3.05	0.07	3.12	0.09
	Average	Average STDEV			Average	Average STDEV	Average	Average STDEV
	3.45	0.15			3.06	0.08	3.14	0.09

All samples in this table have roughly the same composition being based on our Standard LFEP formulation. All of the compositions used a 2.63-2.65 Al/MoO$_3$ molar fuel ratio. The average AUR-AT for the normal primer lots wet loaded was 3.06 $\pm$ 0.08 ms.

The hexane loaded AUR-ATs are on average 0.3 ms better than the dry loaded samples and the standard deviations are also 0.07 ms smaller for the wet loaded. However, this may be the result of either having or not having a post drying step in the pressing procedure. This may be a critical step in the pressing operation. ENC performance is severely degraded by absorption of organic materials and inadequate drying would also degrade LFEP performance. The variation of AUR-ATs for samples that were not dried in the pressing process ranged from 3.11 to 3.98 ms. In comparison, KTHAF144 which was dried gave an AUR-AT almost identical to the wet pressed material. KTHAF144 was the first 100g batch of LFEP prepared and had a slightly deficient AN loading of level of 28.8% instead of 30%.

6.4.3.1 Effects of Different Solvents in Wet Loading Process - The effects of loading LFEPs using Fluorinert (Fluorocarbon solvent) and i-propanol were compared to hexane. Although KTHZ50 does not conform to our standard LFEP formulation with Al/MoO$_3$ molar ratio of 2.50 instead of 2.66, it was used to compare the effects of wet loading with hexane, Fluorinert, and i-propanol solvents. The AUR-ATs collected were 3.33 $\pm$ 0.17, 3.29 $\pm$ 0.29, and 3.58 $\pm$ 0.29 ms, respectively. The Fluorinert FC-77 pressed LFEP performed as well as the hexane while the use of i-propanol gave an AUR-AT that was about 0.25 ms longer. The i-propanol solvent was not pre-dried prior to use and water may have degraded the performance. Fluorinert solvents eliminate the fire hazards associated with hexane and other organic solvents. **Fluorinert solvents could be used in place of water in the LCAAP primer loading process for LFEP. An acceptable environmentally friend fluorocarbon solvent could be used but some modifications would be needed to capture and re-use the solvent.**

6.4.4 New Versus Old Die Set - Although the old die set worked well in the SERDP and early part of the ESCTP programs, the die set was re-designed to press with the ram fitting in the support cup rather than using support cup rim. The original old die set was constructed to match Lake City's die set. The "New" die set was re-designed by China Lake engineers who thought they could improve the design. Two standard batches of LFEP were prepared using Al/MoO$_3$ ENC prepared by the modified ball milling. In Standard LFEP (KTHAD31B) the ENC had been milled for 36 h giving a deflection of 105 mm/g and in Standard LFEP (KTHAD42A) the ENC had been milled for 72 h giving a deflection of 160 mm/g. Samples were pressed dry and wet

with hexane using the **new die set** and fired in the Mann Barrel to collect PVAT data. The ambient AUR-ATs were extremely high for both the dry and wet pressed samples. Standard LFEP (KTHAD31B) gave AUR-ATs of 15 and 32 ms for the dry and wet pressed at ambient. The Standard LFEP (KTHAD42A) gave AUR-ATs of 52 and 63 ms at low-temperature. The consolidation of the LFEP proved to be more difficult with the new die set and smaller amounts of primer was used (140 mg versus 160 mg). The average height of the primers after consolidation is shown in Table 8:

Table 8: Primer Height in Cup After Consolidation Using New Die Set

Weight	Primer Height in Cup	
	KTHAD31B	KTHAD42A
160	0.260	0.263
150	0.260	0.258
145	0.255	0.259
140		0.255
140 (hexane)		0.257
130		0.249
120		0.247

The old die set gave an average height for 160 mg was 0.255 inches. However, the new die set could not meet the height requirement so less LFEP was loaded per round. The maximum load was determined to be 145 mg using the new die set. The low load weight is only partially responsible for the high AUR-ATs. The die redesign puts the entire press load on the bottom of the cup rather than the rim of the cup.

This caused support cup deformation preventing good consolidation (higher primer heights). **The new die set design was abandoned.**

6.4.5 Consolidation of Sonicated Versus Ball Milled Based LFEP - The loose powder density of the Al(80nm)/MoO$_3$(45nm) ENC prepared by modified ball milling is significantly higher (0.59 g/cc) than the small scale sonicated material (0.40 g/cc). However, the ENC density difference had no impact on the primer height after consolidation using the old die set as observed in Table 9:

Table 9: Primer Height in Cup Comparing LFEP Based on Sonication Versus Ball Milling

Primer Wt	KTHAD54	KTHAD56
175		0.255
160	0.253	0.254
155	0.251	0.250
150	0.251	0.247
145	0.244	0.244

Standard LFEP (KTHAD54) used Al/MoO$_3$ made by the small scale sonication process and Standard LFEP (KTHAD56) used the Al/MoO$_3$ from the ball milling process. The LFEP samples were consolidated using the old die set to determine if the consolidation behavior changed due to changes in the scale-up process. The primer heights are nearly identical for both samples regardless of primer weight. The small scale and large scale LFEPs are identical as far as consolidation properties.

6.4.6 Multi-Die Set With Injector Loading - The "Precision Dispensing Systems" from FillPro, Inc. was used to dispense LFEP material into primer cups, dry consolidated and fired on a Mann Barrel gun to determine if the dispensing system and multi-die press would provide similar AUR-ATs to the single loaded rounds. Standard LFEP (KTHAF80) was used in this test. The AUR-ATs for this run were all poor with an average of 3.92 $\pm$ 0.26 ms. At this time, it is not clear why the AUR-ATs for this run were so bad. Not only did the average AUR-AT increase from 3.03 ms to 3.92 but the scatter in AUR-ATs increased as is evident by the larger standard deviation of 0.26 ms. In some wet loading runs, the standard deviation is as low as 0.03 ms. Overall, the multi-die set approach presently requires far too much manual labor and is less efficient than LCAAP current loading & consolidation process. One positive was that the injection loader was demonstrated with the Standard LFEP and greatly reduced loading time. A primer cup loading rate of 700 per hour was achieved as compared to 12 per hour previously. Further testing of the dispensing system is required to determine if the LFEP charges were below 160 mg.

6.5   SAFETY TESTING

Safety test data was collected on scaled-up primers based on the Standard KTHZ60 LFEP formulation. The LFEP was tested dry and as a slurry in hexane. The dry powder had 5 for 5 fires at 10 mJ in the ESD test, had 5 for 5 fires at 50 lbs in the ABL Friction test, and had a 50% point of 33 cm in the Impact test. The Vacuum Thermal Stability test was not performed due to the quantity required for that test (5g). An accidental ignition on a 5 g scale would cause serious damage to equipment and possibly personnel. The ESD, friction, and impact sensitivities were consistent with the LFEP made on a small scale where the ENC was made by sonication. The Safety testing was not performed on the M52A3B1 material by China Lake's Product Quality Laboratory (PQL). The ESD sensitivity for the M52A3B1 primer powders is very high with a reported range of 2 to 98 μJ[28] and ATK has reported 10 for 10 firing of M5A3B1 pellets at 1 mJ. There is no doubt that the M52A3B1 primer would be 5 for 5 fires using China Lake's PQL setting at 10 mJ. The impact sensitivity of lead styphnate has been reported between 2.5 and 5 J and ATK reported the Impact Ignition Energy for the M52A3B1 between 0.2 and 5 J as a function of moisture. The impact sensitivity of the LFEP is consistent with lead-styphnate and the M52A3B1 primer with an onset point of 3.55 J and 50% point of 3.66 J. **In general, the safety properties of the LFEP are very similar to the M52A3B1 primer.** The LFEP/hexane slurry fired 5 for 5 times because the spark ignited the hexane and the slurry burned. The ABL Friction had significantly higher 50% fire point at 417 lbs and onset at 316 lbs. The Impact test for the slurry was 10 for 10 no fires at 200 cm. Burn tests of the LFEP powder (10g) dry and as LFEP/hexane slurry were performed to compare the hazard properties and to obtain an interim DOT classification as a flammable material. As a dry solid, the LFEP produced a loud audio report but did not detonate. The LFEP/hexane slurry burned without exploding. Most safety issues are mitigated by handling of the LFEP materials as slurries.

6.6 CRITICAL PARAMETERS FOR THE Al/MoO$_3$/AN/Kel-F/C LFEP

Al(80nm) - The particle size distribution is critical. Excessive amounts of larger particles result in very poor performing ENCs. The oxide passivation layer thickness should be at least 3.5 -4 nm. Thin oxide passivation layers result in severe continuous aging.

Al(80nm)/MoO$_3$ Molar Ratio – The optimum ratio between 2.63 to 2.69 gave AUR-ATs of 3.06 ms.

Ammonium Nitrate Source – Dyno Nobel's Low-Density AN performs better than other sources. LFEP using Dyno Nobel's AN gave higher Deflections and lower AUR-ATs. The reason for this is not clear but each vendor uses a different proprietary stabilizer coating.

Mixing Sequence – The alternate mixing sequence resulted in > 0.3 ms longer AUR-ATs. Deviations from the standard mixing procedure produced poorer AUR-ATs.

Carbon Addition – Thus far, a functional wet addition approach for carbon has not been identified. Dry mixing is essential at this time.

Dry LFEP – The performance ENCs and LFEPs are drastically reduced by residual organic materials such as organic solvents and oils. Contamination must be avoided and solvents must be completely removed. The dry step is recommended for wet and dry loading procedures.

Die Configuration – The ram should be designed to press using the support cup rim.

LFEP Loading Quantities – A minimum of 165 to 170 mg is needed for low AUR-ATs (< 3.1ms). Lower LFEP loads result in higher AUR-ATs.

6.7 NON-CRITICAL PARAMETERS

Consolidation Pressures – Consolidation pressures between 6500 and 8000 psi gave identical AUR-ATs.

6.8 SUMMARY OF ALL-UP ROUND TESTING

The LFEP gave excellent AUR-ATs results at ambient and low-temperatures for the **Standard LFEP** formulation when the primers were properly prepared. The composition of the **Standard LFEP** formulation was 66% Al(80nm)/MoO$_3$(45nm) ENC, 30% AN, 2% Kel-F, and 2% carbon black. A primer weight load of 160 mg had to be wet loaded, dried, and pressed with our old die set. The Al/MoO$_3$ molar ratio had to be between 2.63 to 2.69 for the nanocomposite component. The carbon had to be dry mixed into the primer not wet mixed. For 4 Standard LFEP batches, Z60, Y64, Y62 and Y50A, the average AUR-AT at ambient and -65°F were 3.03 $\pm$ 0.07 and 3.14 $\pm$ 0.09 ms, respectively, the average barrel pressure was 46.9 Ksi, and average projectile velocity

was 3315 fps. The ranges of AUR-ATs for the Standard LFEP at ambient and low-temperature were 2.89 to 3.15 and 3.05 to 3.27 ms, respectively. Rounds using conventional M52A3B1 lead primers, gave an average AUR-AT at ambient and -65°F of 2.77 $\pm$ 0.10 and 2.96 $\pm$ 0.09 ms, respectively, an average barrel pressure of 47.6 Ksi, and average velocity of 3350fps. The LFEP AUR-ATs are slightly higher than the conventional M52A3B1 primers but they are acceptable. A lower average AUR-AT for the LFEP could be obtained by higher primer load weights. Only 160 mg of LFEP was used in these batches which is the minimum required for good AUR-ATs.

The final SERDP formulation was 76% Al(50nm)/MoO$_3$(45nm), 20% BTATZ, 2% Kel-F, 2%, Carbon Black and gave ambient and -65°F AUR-ATs of 2.98 $\pm$ 0.17 and 3.21 $\pm$ 0.12 ms, respectively, where the goal was less than 4 ms. However, in order for the LFEP formulation to meet the needs of the Navy and Air Force, the action time requirement was changed to MIL-DTL-1394G (AR) Table III where the average action time plus 4 standard deviations cannot exceed 3.5 ms. The F/A-18 and F-22 both use the M61A2 20 mm Gatling gun and the M52A3B1 primer must follow the MIL-DTL-1394G (AR) standard.

The average low temperature AUR-AT + 4 standard deviations for the final SERDP LFEP was 3.69 ms, for the ESTCP LFEP it was 3.50 ms and for the lead based primer it was 3.32 ms. The standard M52A3B1 lead based primer is still the best performing primer available and easily meets the MIL-DTL-1394G low temperature AUR-AT requirements. The current LFEP formulation barely meets the requirements which may pose a problem in acceptance of primer lots during production. Any slight variation in starting materials, LFEP composition of processing could push the lot over the acceptable AUR-AT metric. The current M52A3B1 primer uses 178 mg of Mix FA-874 while 160 mg of LFEP was used in the ESTCP program. However, a higher loading in the range of 165 to 170 mg of LFEP should reduce the AUR-ATs making production more viable. A fact to consider is that the Mil-DTL-1394G (AR) AUR-AT standard may be excessively overdesigned. The M61A2 has a maximum firing rate of 7200 rounds per minute and requires an AUR-AT of 3.5 ms but the M61A2 Gatling gun is currently configured to switch between 4,000 and 6,000 rounds per minute. At a firing rate of 6,000 rounds per minute, a round is fired once every 10 ms and an AUR-AT of 4 ms is adequate. The requirements for the M197 20 mm Gatling gun used in helicopters is must less stringent due to the lower firing rate of 720 rounds per minute. The All-Up Round Action Time Data can be found in Appendix N.

6.8.1 Final All-Up Round 20 mm Testing At Picatinny – The usual NAWCWD process for the fabrication of the 505 20 mm rounds was not used. In general, the LFEP material was prepared, primer cup loaded, pressed and the 20 mm round built-up. Due to funding issues and Base Realignment & Consolidation issues, the normal process was not possible. A large scale batch of the Standard LFEP (KTHAF144 -209g) was prepared on June 9th, 2011. A total of 40 20 mm rounds were made and fired to evaluate the batch within a week of the LFEP preparation. The batch met the 3.5 ms action time requirement. Insufficient funding prevented the loading of the primers and build-up of the 20 mm rounds. The LFEP material was placed in hexane in

polyethylene bottle for safe storage. Funding for the primer loading was secured from the Navy Environmental Sustainability Development to Integration (NESDI) Program (Project 474) for loading and pressing 505 primers and build-up of the 20 mm rounds in Aug. 2011. Support was obtained from PMA 242 to pay for the firing and data collection of the 505 rounds on a M190 Gatling gun. In Sept. 2012, the hexane was removed from the LFEP material and 505 primers loaded and pressed. The primers were completed on Sept. 30th, 2011. Unfortunately, NAWCWD's ammunition capabilities were sent to Picatinny Arsenal in Sept. 2012 and round build-up was not possible at NAWCWD. A contract was established with the Army to complete the round build-up. The 505 primers were sent to Picatinny Arsenal and stored in a magazine until the build-up which occurred in May 2012. The 20 mm rounds were fired in Sept. 2012 but failed to meet the AUR-AT requirements. At this time, the LFEP will not be transitioned by PMA 242. **The firing was a failure.**

Potential Causes of Long AUR-ATs

1. The LFEP material was stored in hexane in polyethylene bottles. The hexane may have leached out stabilizers or other materials from the bottles that ended up in the LFEP when dried.
2. The LFEP may have aged in hexane during the 4 month storage in hexane.
3. The 505 primers were stored in unprotected ammunition boxes in a magazine at Picatinny Arsenal from Nov. 2012 until May 2012 when the rounds were built-up. Aging of the primers could have occurred due to high humidity prior to round build-up (over 6 moths).

6.9   PROJECT SUMMARY

This project had both great successes and bad failures. The LFEP was completely reformulated between the SERDP and ESTCP program. It took 3 years to develop and test the original SERDP formulation. Successes in the ESTCP program include:

1. The LFEP was completely reformulated and re-optimized.
2. The capabilities of the Al Pan Dent Test expanded to test starting materials, intermediate products of the LFEP, and to test the LFEP prior to 20 mm round build up. This test was used to optimize the Al(80nm)/MoO_3 ENC producing the highest deflection numbers at a 2.65 Al to MoO_3 molar ratio. The lowest average AUR-ATs were obtained from formulations using the 2.65 Al/MoO_3 molar ratios in the ENCs. The high performance LFEP samples (low AUR-ATs) all had Deflection numbers of 380 to 550 mm/g and some of the worst performing samples were in the range of 0 to 250 mm/g. The Deflection number for the LFEP was used effectively to screen candidates and monitor run to run variations.
3. New energetic nanocomposites were characterized by the Al Pan Dent Test.
4. A new scale-up process was developed for energetic nanomaterials. A patent has been awarded (US Patent #8231748). This process makes using ENCs in DOD applications

viable for the first time. Two other patents awarded on the desensitization of MIC (US Patent # 7789981 and 7931764).

5. A scale-up process was developed for the LFEP.
6. A powder injector system for loading into primer cups demonstrated using the LFEP.
7. Critical parameters affecting the AUR-ATs were identified including Al/MoO_3 molar ratios, LFEP composition, LFEP formulation sequence, and consolidation factors.

The failures of the ESCTP program severely impacted scheduling, budgeting and completing tasks. These failures include:

1. Management failure to monitor funding expenditures.
2. Management failure to monitor tasks and completions of tasks on schedule.
3. Management failed to adequately document and update primer loading and consolidation procedures. The processes changed over time leading to erratic AUR-ATs. Process reviews had to go back and determine changes made and identify sources of the problems created by the changes.
4. Poor management decisions and utilization of funds. The standard die set was re-designed even though the original die configuration was producing good results. This was an unnecessary expenditure of funds that led to program delays. The die set was re-designed, fabrication of the die sets took a year, and the new die sets resulted in much higher AUR-ATs. A series of 20 mm rounds had to be made to determine if the new die set was at fault or if it was the LFEP. The management team and the die set designers believed it was the LFEP not the new die set. The new die set and testing delayed the program back at least 1.5 years. Management also decided to design and build a stainless mixer for the final scale-up step of the LFEP where carbon black had to be dry-mixed. This process required hundreds of man hours in design, safety reviews (5 reviews with 20 + people for 3 + hrs each), mixer construction and a mixing run. The AUR-AT for this run was horrendous with action times ranging from 4 to 50 ms. Ultimately, 2 100g runs were completed in the laboratory in 6 man hours. This decision cost the program in funds but also led to a 4-5 month delay. The decision to build a complex multi-die set that pressed a 2D array was expensive and unwarranted. LCAAP had a fast automated pressing system where a 5 by 5 array of primers are pressed one 5 primer row at a time, the tray moved and the next row pressed. This process is automated and fast. In comparison, the multi-die set requires manual assembly of a complex multi-plate system. The multi-die system required complex engineering to build and was too labor intensive to replace the automated system used by LCAAP. Ultimately, the program ran out of funds without completing a number of tasks including aging study of the scaled-up LFEP, Gatling gun testing of the scaled-up LFEP, meeting with transition partners Energetic Materials & Products Inc. (EMPI), General Dynamics-Ordnance and Tactical Systems (GD-OTS), and LCAAP, setting up qualification for DOD use, completing a cost assessment and without writing a final report.

6.91 Lessons Learned

1. Continuous personnel changes especially the PI are disruptive and should be avoided.

2. Engineering redesigns to improve functional equipment is not acceptable.

3. Schedule, expenditures, and tasks must be closely monitored by the PI.

4. The starting materials should be characterized and material specifications should be developed when a formulation is chosen.

5. A consistent process for building up rounds should be used, maintained, and changes documented via changes in the SOP throughout the project.

7.0 COST ASSESSMENT

7.1 COST MODEL

Several cost models are reported including the actual scale-up and fabrication of 505 rounds for final all-up round testing, a cost model using a new powder injector for the LFEP primer cup loading, a cost projection of 505 primers using a slightly modified LCAAP primer loading process, a cost projection of a production run of 100,000 rounds at LCAAP based on the high current cost of 80 nm Al, and a cost projection of a production run of 100,000 rounds at LCAAP based on the future projected price of 80 nm Al.

For the all runs, the preparation of the LFEP is based on the scale-up method developed by China Lake. The LFEP is made by a modified ball milling of 80 nm Al and MoO_3 in a solvent, followed by the addition of AN, and Kel-F. The solvent is removed, carbon is added and dry mixed. Fresh solvent is added to the LFEP and the milling balls are removed by pouring through a sieve. Excess solvent is removed to produce a paste or dry powder.

In the NAWCWD Standard Method, 160 mg of the LFEP powder was weighed out, hexane added to make a slurry, the slurry placed in the primer cup, the hexane evaporated off and the primer pre-pressed at 200 psi, the primer dried at 60°C for 4 hours, and then consolidated at 8000 psi. Each primer was processed individually leading to higher costs. NAWCWD also has a 2 man rule for safety which doubled the man hours required to perform the loading & consolidation operations.

In the NAWCWD Process using the powder injector, the LFEP was injected into primer cups in a 5x5 tray. The injector was set to 165 mg. The injector enabled a LFEP loading rate of 12 cups per minute. The multi-die set system was used as described in Section 5.1.3.3.

For a minimum impact on LCAAP primer loading process where Mix-874 is loaded as a water based paste, a slightly modified process could be used. Water is not a viable solvent for the China Lake LFEP but the fluorocarbon liquid, Ethyl Nonafluoroisobutyl Ether (HFE-7200), could be used to replace water. HFE-7200 is environmentally friendly, HFE-7200 is not a fire hazard, and the LFEP/HFE-7200 paste should be safer to handle. Solvent recycling systems are required to minimize costs.

The cost for 505 LFEP was estimated based on using LCAAP's standard primer loading process but using a solvent recovery system in place of LCAAP's open oven for the drying step. The cost of a 100,000 LFEP run was also estimated. A final cost model was based on the price of the 80 nm Al dropping down to $100/Kg which is NovaCentrix's future cost target.

7.2 COST ANALYSIS AND COMPARISON

The NAWCWD standard method was labor intense where primer cups were loaded one at a time and the consolidation process was one at a time. In addition, a 2 man policy was in effect where a safe man had to be present at all energetic operations. The labor rates at NAWCWD are also higher than what is expected for a manufacturing environment such as Lake City Army

Ammunition Plant (LCAAP). The cost of producing LFEP was calculated using three scenarios. The first scenario, NAWCWD Standard Method, was the actual costs associated with preparing 505 primers that were used to build 505 20 mm rounds for testing at Picatinny Arsenal. The primers were individually made. The LFEP was loaded as hexane slurry into a primer cup, paper disk added, support cup pressed in, the primer cup dried in an oven, and then pressed at 7500 psi. This process cost $38 per primer. In the second scenario, the primer cups were filled using a new powder injector that had been calibrated to deliver 160 mg of dry LFEP powder into each cup. The paper disk and support cup was added and the primer pressed at 7500 psi. This led to a dramatic decrease reducing the cost per primer down to $13 per primer. In scenario three, LCAAP 100,000 Round LFEP run, primer balls are made out of the LFEP and HFE-7200. HFE-7200 is a relatively benign solvent that has a Global Warming Potential of 55, non-toxic by ingestion, no skin contact issues, and has a relatively high inhalation exposure limit of 49,000 ppm. The high cost of $74/Kg warrants recycling by using a solvent re-cycling system. This approach would enable LCAAP to load and consolidate the 20 mm primers with minimal change to their process. Using the high current cost for 80 nm Al of $2500 per Kg, the cost per LFEP is $0.47 which is comparable to the standard M52A3B1 primer ($0.45 estimated). In the forth scenario, the cost of the 80 nm Al has been reduced to $100 per Kg. NovaCentrix believes that the cost for 80 nm Al could be reduced to $100 per Kg in a continuous production mode where at least 40 Kg per month is made. The sales price would be higher than $100/Kg but $100/Kg was used in this cost model. This reduced the price per LFEP down to $0.35. The cost for producing the standard M52A3B1 primer was also calculated based on educated guesses. LCAAP would not divulge the costs associated with its production but was calculated to be around $0.45 per primer. The cost scenarios are summarized in Tables 10 & 11

Table 10: Actual costs to produce LFEP for 20 mm rounds

Task	NAWCWD Standard Method 505 Rounds		NAWCWD Using Powder Injector 505 Rounds	
	Cost	Man Hours	Cost	Man Hours
Preparation of LFEP (83.5g)				
Materials Al, MoO$_3$, AN, Kel-F, C, hexane	59		59	
Labor	300	2 hr @ 150	300	2 hr @ 150
LFEP Loading				
Primer cup, paper disk, support cup	25		25	
Labor	11220	102 hr @ 110	220	2 hr @ 110
LFEP Consolidation	7480	68 hr @ 110	5500	50 hr @ 110
Inspection	440	4 hr @ 110	440	4 hr @ 110
Total cost for primers	19524		6544	
Cost per primer	**$38.66**		**$12.96**	

Table 11: Cost Projections for Lead-Free Primers at LCAAP

Task	LCAAP 505 Round Run $2500/Kg for Al		LCAAP 100,000 Round Run $2500/Kg for Al		LCAAP 100,000 Round Run $100/Kg for Al		Standard LCAAP M52A3B1 100,000 Round Run
	Cost	Man Hours	Cost	Man Hours	Cost	Man Hours	
Preparation of LFEP (83.5g)							
Materials Al, MoO_3, AN, Kel-F, C, hexane	59		11647		1509		1000
Labor	160	2 hr @ 80	640	8 hr @ 80	640	8 hr @ 80	8800
LFEP Loading							
Primer cup, paper disk, support cup	25		5000		5000		5000
Labor	160	2 hr @ 80	12800	320 hr @ 80	12800	320 hr @ 80	12800
LFEP Consolidation	160	2 hr @ 80	12800	320 hr @ 80	12800	320 hr @ 80	12800
Inspection	24	0.3 h @ 80	4480	56 h @ 80	4480	56 h @ 80	4480
Total cost for primers	588		47367		37229		44880
Cost per primer	**$1.16**		**$0.47**		**$0.37**		**$0.45**

Total Capital Equipment Costs							$83,000
Installation							15000
Milling Equipment – 3 Mills, Milling media							13000
Equipment – 2 Hoods, 2 Solvent Recycling Systems							35000
HFE-7200 Solvent (600 lb Barrel)							20000

7.2.1   Facilities Capital Cost – The initial capital expenses for new equipment and installation is $83,000 if transitioned to Lake City. This cost includes the purchase of 3 US Stoneware Model 784 Unitized Jar Mills, 500 lbs of 5 mm stainless steel milling balls, 1 vacuum oven with solvent trap for primer drying (pre-pressing), 1 solvent recycling system for LFEP material synthesis, 1 hood for primer ball storage & handling, 1 hood for solvent additions, and the purchase of HFE-7200 solvent. Items such as hoods and milling equipment may or may not be currently available at LCAAP, so the costs were included. At a new production facility, costs will be significantly higher. Additional equipment that is needed include an automated press, automated shakers that put the support cups in the primer cup, work benches, grounded work stations, computerized inspection stations, and automated system that fill primer boxes with finished primers. The cost of the equipment probably exceeds $1M.

7.2.2   Start-up cost – New standard operating procedures (SOP) and training are needed prior to start up. Although the properties of the LFEP are comparable to the Mix FA-874, the preparation process is different. All the LFEP preparation processes are mixing processes that give high yields and very little waste. However, it is expected that the solvent used, either hexane or HFE-7200, will be recycled not discarded as hazardous waste. The development of an approved SOP is expected to cost $40,000 (500 hr x $80). Training of personnel is estimated at $6400 (4hr x 20) annually.

7.2.3   Operations and maintenance cost – The maintenance cost of the Unitized Jar Mills is expected to be relatively low. The rollers wear out over time and should be replaced every 5 years but the wear will be dependent on usage. There are 36 rollers that will need periodic replacement (36 x $50 = $1800 or $300/yr + 4 hr x $80 (labor) = $320). The Unitized Jar Mills are expected to have a service life of at least 15 years and a replacement cost of $1K per year. The high density polyethylene bottles used in the milling process will cost less than $1000 per year. The recycling of the solvent HFE-7200 will not be 100%. A loss of 1 Kg (12% loss) of HFE-7200 per 100,000 primer run will cost $75 per run or $1500 per year based on 200 runs. Overall, the cost of maintaining and replacing components is expected to be about $5000 per year. The maintenance cost of Lake City's current system is not known but is probably significantly higher. To treat LFEP waste & equipment, either a weakly basic water solution or a 2% solution of Simple Green can be used. This generates a waste stream with aluminum hydroxide, molybdenum oxide, Kel-F, and carbon in water.

7.2.4   Occupational Health, Safety, Hazardous Waste, & Monitoring – A major cost benefit is the elimination of the lead and barium waste streams. These waste streams cost Lake City an estimated/guess $50K per year. As a consequence, health monitoring for lead can be eliminated saving an estimated/guess $30K per year. A major unknown consideration is the effects of exposure of workers to nano aluminum and nano molybdenum trioxide powders. Neither of these materials is easily aerosolized and the particles tend to agglomerate into large multi-micron sized aggregates. This behavior is expected to reduce the inhalation risk. The chief exposure to these powders is during the weighing and loading processes. Automated dispensing technology could be used to eliminate exposure during weighing however loading the Al into the dispensing unit could still be an issue. Future regulations on the use of nanosized powders are likely but may not impact all nanomaterials.   The DOD will benefit from the elimination of lead in DOD primers at indoor and outdoor ranges, by eliminating exposure of soldiers to airborne lead in practice exercises and in the field, and by eliminating a source of lead that contaminates the air,

water, soil, flora and fauna.   Future savings are anticipated from indoor and outdoor ranges when lead projectiles and primers are eliminated from DOD ammunition. Workers exposed to lead at a level of 30 μg/m^3 for 30 days per year require blood lead level monitoring. OSHA requires medical removal from exposure at blood lead levels of 50 μg/dL. Lead monitoring will not be required once the DOD removes lead from its ammunition and remediates its ranges. In addition, future range shut downs due to lead contaminated ground water will be eliminated.

8.0 IMPLEMENTATION ISSUES

8.1   STAKE HOLDER

Currently, PMA-242 is funding the final testing of the 505 Scaled-Up 20 mm rounds to be fired at Picatinny Arsenal in Sept. 2012. The rounds will be fired in a M197 gun and if successful, PMA-242 will submit the LFEP to the Weapons System Explosive Safety Review Board (WSESRB) for qualification. The WSESRB will generate actions required for qualification and usually requires about 12-18 month to collect the required data. Picatinny/Crane will provide the engineering and testing support for explosive qualification requirements and CL will provide the Safety Officer (John Alves) as the representative for WSESRB. Qualified product means it is safe for use by N/MC. Following or concurrent to the WSESRB, PMA-242 would generate an Engineering Change Proposal (ECP) for 20MM ammunition using LFEP as a suitable substitute for NAVAIR 20MM PGU and the Army's M940 and M56 ammunition. Approved ECP means it would be produced by the industry for the fleet. Cost to implement into industry could be included in the ECP OR would be done by the industry and covered in an increased cost per round. PMA-242 and the Single Manager for Conventional Ammunition (SMCA) would be responsible for the requirements. The Joint Munitions Command has to buy into the changes and SMCA would execute the contract modifications.

8.2   TECHNOLOGY TRANSFER

The scale-up processes for the LFEP has been protected by the US government by 3 patents. The technology would be licensed to the industry for ammunition production. NAWCWD as the subject matter expert would support the manufacturing and production of the LFEP based ammunition. The additional equipments needed are commercial off the shelf items and should not hinder the transition to LFEPs.

8.3   ENVIRONMENTAL CHECKLIST

The regulation of nanomaterials by states and the EPA could impact the production and use of the LFEP. At this time, there are no US or international laws regulating nanomaterials. The Project of Emerging Nanotechnologies established in 2005, lists over 800 nanotechnology products that are currently unregulated. However, new laws and regulations may be coming in the future. The Department of Toxic Substances Control initiated the second Chemical Information Call-in for six nanomaterials: nano cerium oxide, nano silver, nano titanium dioxide, nano zero valent iron, nano zinc oxide, and quantum dots. The Call-in requested further information of analytical test methods, fate and transport in the environment, and other relevant information under California Health and Safety Code, Chapter 699, sections 57018-57020.

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Dixon, Phil

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Federal Cartridge Company
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Pritham, Charles H., Darby, Upper, Rechel, Ernest, and Stevenson, Thomas
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US Patent No. 7,931,764

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Dreizin, E. L. and Schoenitz, M.
US Patent No. 7,524,355

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Kelvin T. Higa
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Billon, H., Redman, L.
DSTO Technical Report DSTO-TR-0029, Sept. 1994

APPENDICES

Appendix A: Points of Contact

Point of Contact	Organization	Phone/Fax/ email	Role in Project
Eric Gogley	NAWCWD Code 478200D 2400 E Pilot Plant Road China Lake, CA 93555	760-939-7664	Principal Investigator
Kelvin Higa	NAWCWD Code 2L4200D 1900 Knox Road, Stop 6303 China Lake, CA 93555-6106	760-939-1656	Research Chemist
Donald Herigstad	NAWCWD (Jacobs Technology)	760-939-7507	Associate Investigator
Rao Yalamanchili	ARDEC	973-724-2487	US ARMY/Engineer
Timothy Foley	LANL Los Alamos National Lab. C-ADI, Mail Stop J565 PO Box 1663 Los Alamos, NM 87545	505-665-3583	Research Chemist
Pierre Lemay	GDOTS Canada Inc.	514-806-9827	Industry Consultant
Karl Martin	NovaCentrix	512-491-9500	Industry Consultant
Dan Clark	LCAAP	816-796-5228	Industry Consultant

Appendix B: The "Al Pan Dent Test"

The Al Pan Dent Test was initially developed at NAWCWD for the rapid evaluation of nano-energetic composites. It quickly became apparent that this test could be used for a variety of rapid testing and optimizations. The Al Pan Dent Test can be used for:

1) New NEC Development
2) NEC Optimization
3) Starting Material Quality Control
4) Effects of Additives (Gas, Binder, Metals, Carbon, Thermites, ..)
5) LFEP Optimization

Materials

Sigma-Aldrich	Disposable Aluminum Dishes (Z154849)
Electro Technic Products, Inc.	Tesla Coil Model BD-10A
Aluminum Plate	
Scherr-Yumico Inc	Micrometer

Warning: When testing nano energetic composites, all equipment and operators should be electrically grounded, and personal protection equipment should include safety glasses, laboratory coat, ear protection and a grounded wrist strap.

General Procedure

1. The test material is broken down into a fine powder using a grounded spatula on a grounded piece of Al foil.
2. An Al Pan (Aluminum Dish) is placed on a grounded Analytical Balance (preferably accurate to 0.0001 g) and zeroed.
3. The test material is placed in the center of the pan and the pile size minimized using the grounded spatula.
4. The sample is transferred to the Tesla Coil setup shown in the figure below:
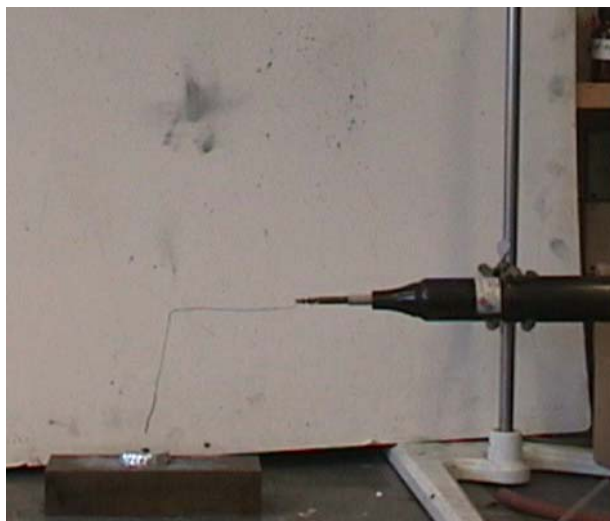
Figure B.1: Al Pan Dent Test Set-Up

5. The Tesla Coil is equipped with a metal wire which acts as an electrode. The tip of the electrode should be about 0.25 to 1.0 inches over the test material. In the figure, a metal block is used as the ground but it is not required. The Tesla Coil is set on maximum to ensure reproducibility. This test is performed in a hood to prevent inhalation of by-products.
6. The Tesla Coil is turned on that generates a spark igniting the test material.

7. The Al Pan is placed on the Al Plate inverted and the dent + Al Plate + Al Pan height is measured using a micrometer. The dent height is calculated by subtracting the Al Plate Thickness and Al Pan Thickness. The "Deflection" is calculated by dividing the dent height divided by the weight of the sample and reported in mm/g.

Note: The "Deflection" number will be sensitive to the Al Pan thickness so the use of other brands of Al Pans will produce slightly different results. The quantity of test material used should be kept constant ($\pm$ 1 mg). Some materials tested show little deviation in Deflection with weight while other samples vary significantly. Large agglomerated particles in the test sample will produce lower Deflection numbers so should be avoided.

Validation of the "Al Pan Dent Test"
Traditionally, nano energetic composites (MIC, Super-Thermite) have been optimized by pressure cell testing by LANL and various universities. This is a time consuming process that is not well suited for rapid analysis. A series of NECs were optimized by the Al Pan Dent Test and compared to the same materials optimized by LANL's pressure cell. A strong correlation was found between the Deflection and LANL's peak pressure from their pressure cell. A plot of the optimization of Al(80nm)/MoO$_3$(45nm) by LANL and by the Al Pan Dent Test is shown in the figure below:
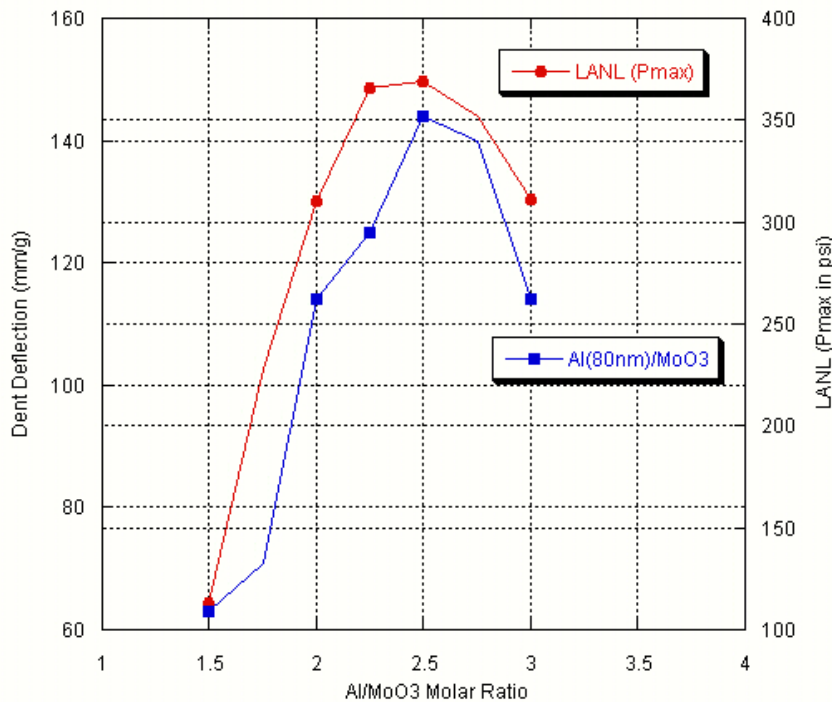
Figure B.2: Optimization of Al(80nm)/MoO$_3$ by Pressure Cell Versus Al Pan Dent Test

The optimizations of several NEC are depicted in Figures B.3 to B.9 and many have not been optimized by the conventional pressure cell method. In Figure B.3, an 80 nm Al batch M2453 was optimized using the Al Pan Dent Test. Most 80 nm Al batches optimized between 39 to 41 weight percent Al compositions with the MoO$_3$ oxidizer.

70
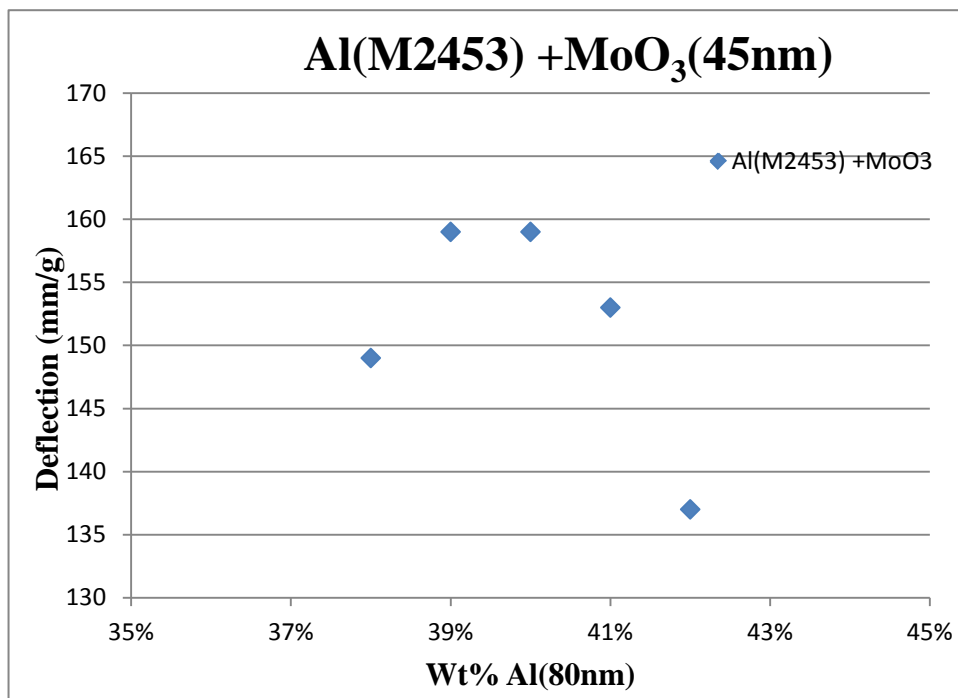
Figure B.3: Optimization of Al(80nm)/MoO$_3$ Using Lot M2453

Figure B.4 shows the optimization of the Al(80nm) and CuO ENC. The stoichiometric ratio for this composite in 0.67 Al to CuO but high performance is seen for a wide range of Al/CuO ratios.
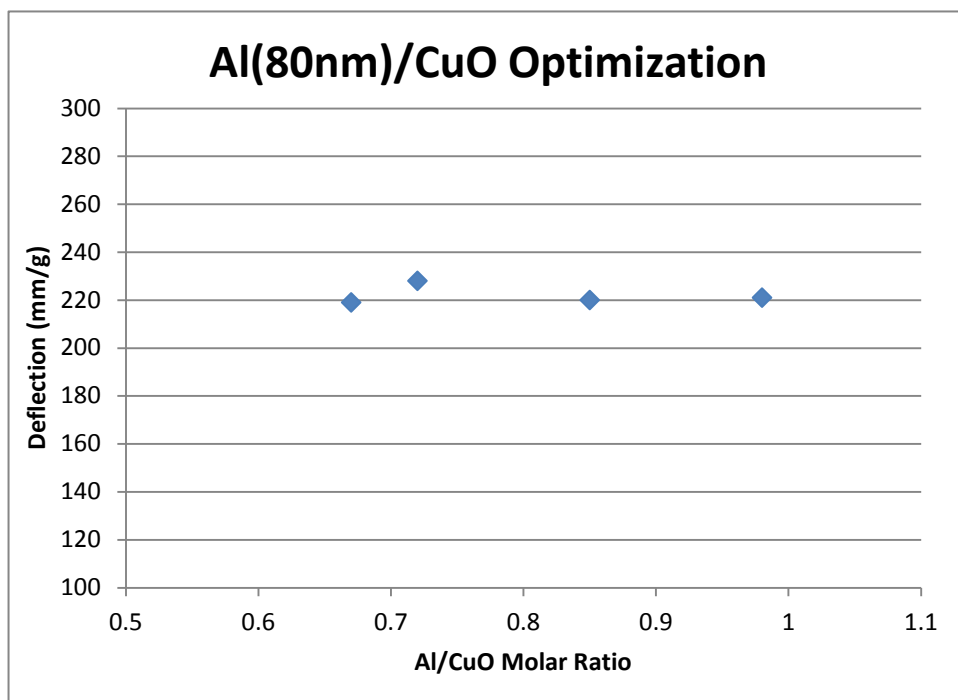
Figure B.4: Optimization of Al(80nm)/CuO(40nm) ENC

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Figure B.5 shows the optimization of Al(80nm) and Bi_2O_3(320nm) ENC. High performance was observed at 15 weight percent Al that corresponded to a 2.25 to 1 Al to Bi_2O_3 molar ratio. This material is one of the most ESD sensitive materials tested to date. Extreme caution should be used in the preparation and handling.
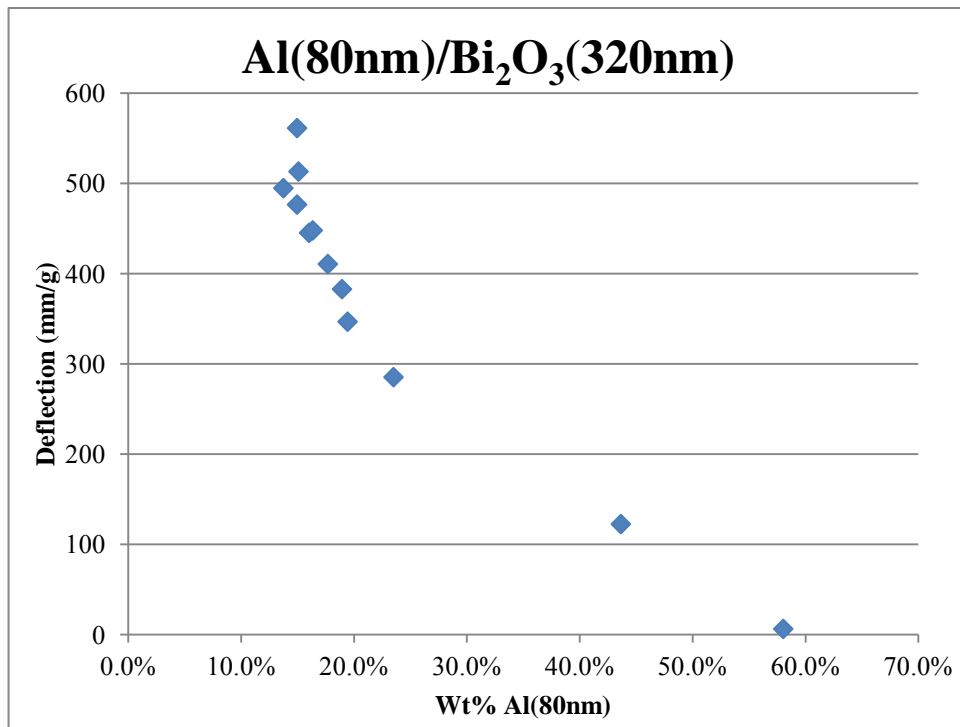
Figure B.5: Optimization of Al(80nm)/Bi_2O_3(320nm)

Figure B.6 shows the partial optimization of Al(80nm) with a 2.5 micron sized Bi_2O_3 powder. A maximum deflection of about 105 mm/g was observed at 12 weight percent Al which corresponds to a stoichiometric 2 to 1 Al/Bi_2O_3 molar ratio. However, a ratio less than 2 was not tested. The performance of this composite is very low due to the large Bi_2O_3 particle size.
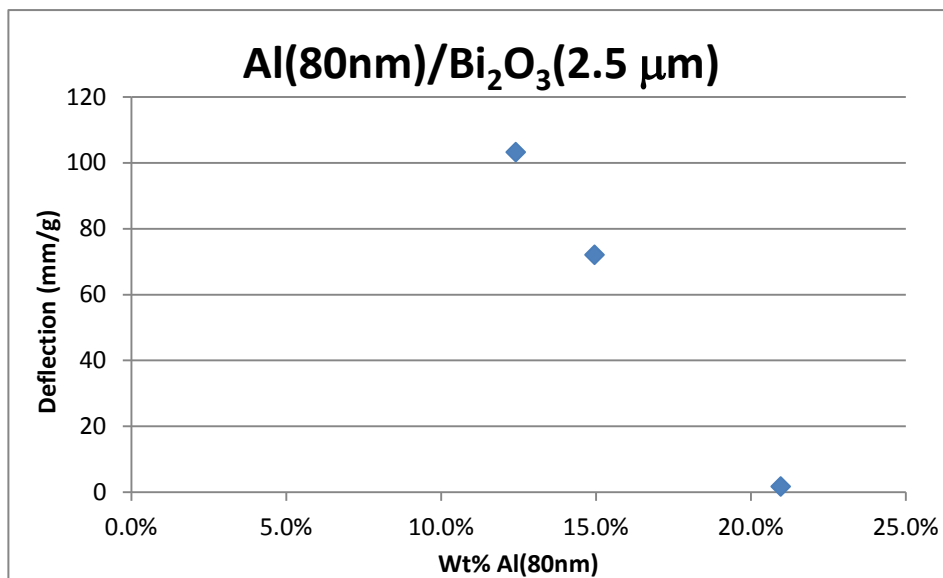
Figure B.6: Optimization of Al(80nm)/Bi$_2$O$_3$(2.5 μm)

In Figure B.7, 80 nm Al and 150 nm (ALEX) with 320 nm Bi$_2$O$_3$ were optimized to compare their overall performance. The ENCs made with the significantly cheaper ALEX powder were nearly as good as composites made with 80 nm Al. For most applications using the Al/Bi$_2$O$_3$(320nm) ENC, ALEX would be the preferred Al source.
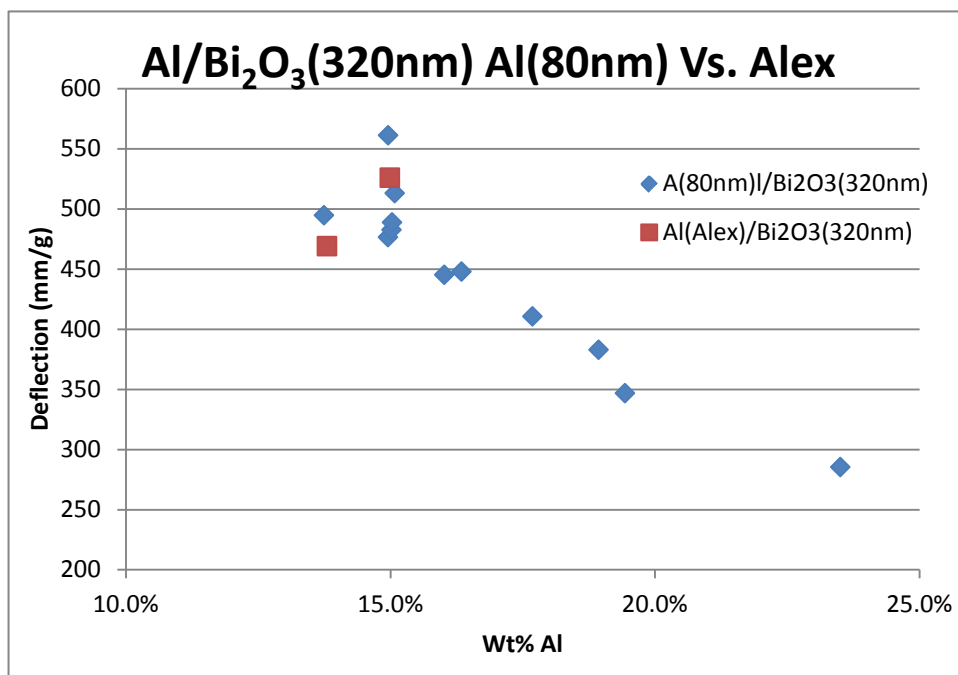
Figure B.7: Al/Bi$_2$O$_3$(320nm) Al(80nm) Versus ALEX

In Figure B.8, the new ENC Al/Bi(IO$_3$)$_3$ was optimized. The stoichiometric ratio of Al to Bi(IO$_3$)$_3$ is 6 to 1 however, very high performance is observed between 7 to 9. This composite is the second best

performing composite observed to date, only exceeded by the Al/AgIO$_3$ ENC. One advantage is that silver is not used in the formulation negating the use of costly silver and avoiding potential environmental issues associated with silver.
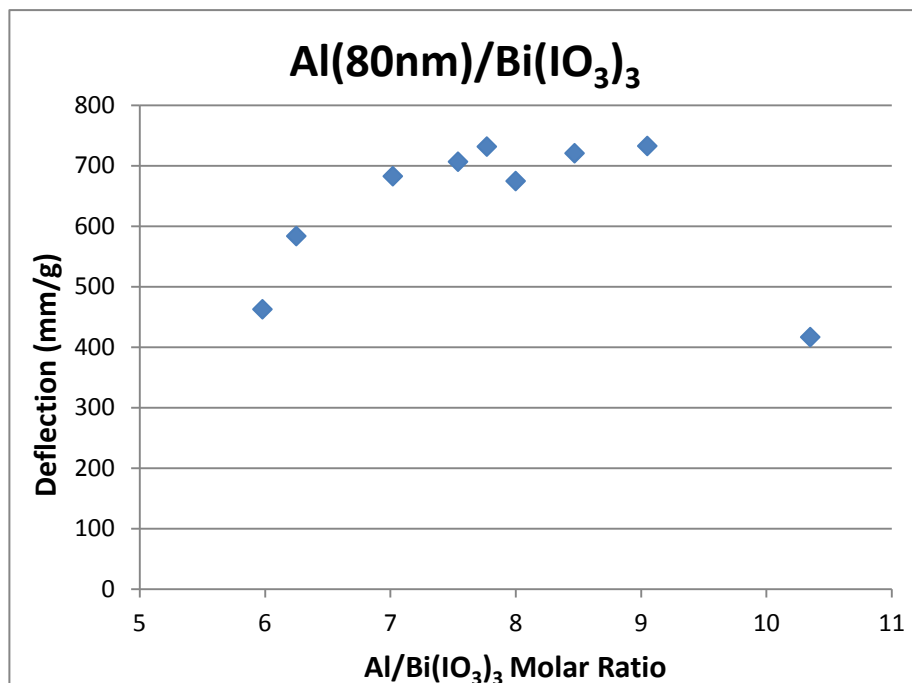
Figure B.8: Optimization of Al(80nm)/Bi(IO$_3$)$_3$

In Figure B.9, the Al(ALEX)/MoO$_3$(45nm) composite was optimized by ball milling the composite. The usual preparation by ultra-sonication led to very poor results with deflections under 100 mm/g. The Al(ALEX)/MoO$_3$(45nm) composite optimized at 37 weight percent ALEX which corresponded to a 2.68 to 1 Al to MoO$_3$ molar ratio. This matches the optimum Al/MoO$_3$ molar ratio for 80 nm Al. The Deflection for the sonicated sample was only 76 mm/g as compared to 224 mm/g for the ball milled.
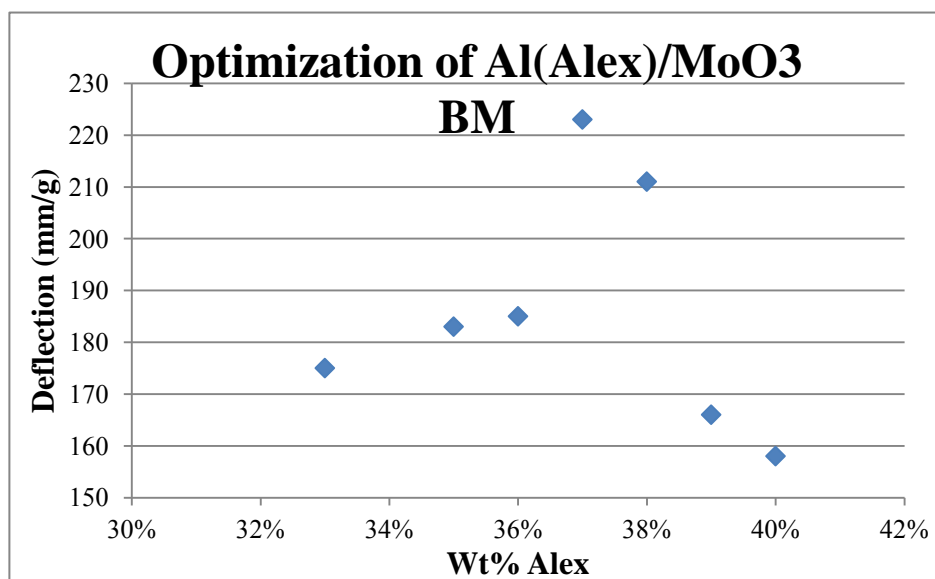
Figure B.9: Optimization of ALEX/MoO$_3$(45nm) Prepared by Ball Mill

Appendix C: MIL-DTL-1394G, Military Specification, Primers, Medium Caliber Ammunition

MIL-DTL-1394G(AR)

3 MAY 2006_____

SUPERSEDING

MIL-P-1394F(AR)

23 November 1983

DETAIL SPECIFICATION

PRIMER, ELECTRIC: M52A3B1

> Reactivated after 3 May 2006 and may be used for new and existing designs and acquisitions.

This specification is approved for use by all Departments and Agencies of the Department of Defense and is available for use by all Departments and Agencies of the Department of Defense.

1. SCOPE

1.1 Scope. This specification covers the electric primer M52A3B1, for use in the assembly of 20mm ammunition (see 6.1).

2. APPLICABLE DOCUMENTS

2.1 General. The documents listed in this section are specified in sections 3 or 4 of this specification. This section does not include documents cited in other sections of this specification or recommended for additional information or as examples. While every effort has been made to ensure the completeness of this list, document users are cautioned that they must meet all specified requirements of documents cited in sections 3 and 4 of this specification, whether or not they are listed.

2.2 Government documents.

2.2.1 Specifications, standards and handbooks. The following specifications, standards and handbooks form a part of this document to the extent specified herein. Unless otherwise specified, the issues of these documents are those cited in the solicitation or contract (see 6.2).

DEPARTMENT OF DEFENSE SPECIFICATIONS

MIL-A-70625 - Automated Acceptance Inspection Equipment

Design, Testing and Approval, of

DEPARTMENT OF DEFENSE STANDARDS

MIL-STD-1916   - DOD Preferred Methods for Acceptance of Product

MIL-STD-1168 - Ammunition Lot Numbering and Ammunition Data Card

  Comments, suggestions, or questions on this document should be addressed to: Commander, U.S. Army ARDEC, ATTN: AMSRD-AAR-AIS-SS, Picatinny, New Jersey 07806-5000, or ardec-stdzn@pica.army.mil. Since contact information can change, you may want to verify the currency of this address information using the ASSIST online database at http://assist.daps.dla.mil.

METRIC

 2.2.2 Other Government documents, drawings and publications. The following other Government documents, drawings and publications form a part of this document to the extent specified herein. Unless otherwise specified, the issues of these documents are those cited in the solicitation or contract.

U.S. ARMY ARMAMENT RESEARCH, DEVELOPMENT, AND ENGINEERING CENTER (ARDEC) PUBLICATIONS

SCATP-20 - Ammunition Ballistic Acceptance Test Methods,
    Test Procedures for 20mm Cartridges

(This publication is available from US Army ARDEC, AMSRD-AAR-QEM-F, Picatinny, NJ 07806-5000.)

U.S. ARMY ARMAMENT RESEARCH, DEVELOPMENT, AND ENGINEERING CENTER (ARDEC) DRAWINGS

7548057   Cup, Primer
7548058   Button, Primer
7548066   Primer, Electric, M52A3B1 Assembly
7258817   Cartridge, 20mm, Target Practice, M55A2
7548108   Cup, Support, Primer
1575AS300 Cartridge, 20mm, Target Practice, PGU-27/B

(Copies of these drawings may be requested on line at Drawing-Request@pica.army.mil or from US Army ARDEC, AMSRD-AAR-AIS-TD, Picatinny, NJ 07806-5000.)

ARMY MATERIAL COMMAND PUBLICATIONS

ORD-SIP-S314 - Visual Inspection Standards for Small Arms

Ammunition Primer Defects

(This publication is available from US Army ARDEC, AMSRD-AAR-QEM-F, Picatinny, NJ 07806-5000.)

2.3 <u>Order of precedence</u>. In the event of a conflict between the text of this document and the references cited herein, the text of this document takes precedence. Nothing in this document, however, supersedes applicable laws and regulations unless a specific exemption has been obtained.

3. REQUIREMENTS

3.1 <u>First article</u>. When specified, a sample shall be subjected to first article inspection in accordance with 4.2.

3.2 <u>Parts and Subassemblies.</u> Materials, parts and assemblies shall comply with requirements specified on the applicable drawings and referenced specifications.

3.3 <u>Insulation strength</u>. The cup-insulator-button assembly shall withstand an electrical potential as specified on drawing 7548066.

3.4 <u>Electrical resistance</u>. The electrical resistance of the primer assembly (dry) shall be as specified on drawing 7548066.

3.5 <u>Pellet weight</u>. The total dry weight of the primer mix shall be as specified on drawing 7548066.

3.6 <u>Electric primer sensitivity</u>. The primer shall function when energized by a 10 microsecond discharge from a 2 microfarad condenser charged at 160 volts DC.

3.7 <u>Electric primer time</u>. The primer time shall not exceed 300 microseconds when energized by a 10 microsecond discharge pulse from a 2 microfarad condenser charged at 160 volts DC.

3.8 <u>Action time</u>. The action time of the test cartridge shall be as specified on drawing 7548066.

3.9 <u>Function and casualty</u>. The primer shall function without casualty in the test cartridge (see Table VII).

3.10 <u>Workmanship</u>. All parts and assemblies shall be fabricated, loaded, and assembled in a thorough workmanlike manner. They shall be clean and free of burrs, sharp edges, cracks, scratches, dents, folds, wrinkles, buckles, dirt, grease, oil, rust, and other foreign matter. Exterior

surface coatings shall be continuous; however, light scratches not exposing base material may be permitted.

4. VERIFICATION

TABLE I. Requirement/verification cross reference matrix

 METHOD OF VERIFICATION CLASSES OF VERIFICATION

 N/A - Not applicable   A - First article

1 - Analysis   B - Conformance

2 - Demonstration

3 - Examination

4 - Test

Section 3 Requirement		Verification Methods					Verification Class		Section 4 Method
		N/A	1	2	3	4	A	B	
3.1	First article				X	X	X		4.2
3.2	Parts and subassemblies					X	X	X	Table IV
3.3	Insulation Strength					X	X	X	4.4.1
3.4	Electrical resistance					X	X	X	4.4.2
3.5	Pellet weight				X		X	X	4.4.3
3.6	Electric primer sensitivity					X	X	X	4.4.4
3.7	Electric primer time					X	X	X	4.4.5
3.8	Action time					X	X	X	4.4.6
3.9	Function and casualty					X	X	X	4.4.7
3.10	Workmanship				X		X	X	Table IV

4.1 <u>Classification of inspection</u>. The inspection requirements specified herein are classified as follows:

a. First article inspection (see 4.2)

b. Conformance inspection (see 4.3)

4.2 <u>First article inspection</u>. When specified, a sample of 2000 M52 primers and primer components as identified in Table II shall be subjected to first article verification inspections and tests with quantities in accordance with Table II and Table IV.

4.2.1 <u>First article rejection</u>. If any assembly, component or test specimen fails to comply with any of the applicable requirements, the first article sample shall be rejected.

TABLE II. First article inspection

Examination or Test	Conformance Criteria		Requirement Paragraph	Inspection Method	Defect Classification
	Sample	Acc/ Rej			
Examination for defects					
Cup, primer	125	0/1	3.2/3.10	Table IV	Table IV
Button, primer	125	0/1			
Cup, support, primer	125	0/1			
Primer, electric	2000	0/1			
Insulation Strength	2000	0/1	3.3	4.4.1	Major
Electrical resistance	2000	0/1	3.4	4.4.2	Major
Pellet weight	2000	0/1	3.5	4.4.3	Critical
Electric primer sensitivity <u>4</u>/	800	0/1	3.6	4.4.4	Major
Electric primer time <u>5</u>/	50	0/1	3.7	4.4.5	Critical
Action time -65°F <u>6</u>/	50	0/1	3.8	4.4.6	Critical
Function and casualty	1100	0/1	3.9	4.4.7	Major

See Notes after Table III

4.3 Conformance verification.

4.3.1 <u>Conformance inspection</u>. The sample cartridges shall be subjected to conformance verification in accordance with Table III and Table IV.

 4.3.2 <u>Classification of characteristics</u>. Critical, major and minor characteristics are defined in MIL-STD-1916.

 4.3.3 <u>Inspection lot formation</u>. Lot formation shall be in accordance with MIL-STD-1916. Lot numbering shall be in accordance with MIL-STD-1168.

  4.3.4 <u>Conformance rejection</u>. If any sample fails to comply with the conformance inspection requirements, the lot shall be rejected.

 4.3.5 <u>Examinations and tests</u>. The attribute sampling plan required for the examination for defects in Table IV shall be in accordance with the attribute sampling plan of MIL-STD-1916, using Verification Level IV for major characteristics and Level II for minor characteristics unless otherwise noted. One hundred percent inspection shall be used on all critical characteristics. The lot shall be suspended if a malfunction or casualty not covered by this specification occurs in any firing test (see 6.9).

 4.3.6 <u>Alternative conformance acceptance</u>. Unless otherwise specified, alternate conformance procedures may be proposed (see 6.2).

TABLE III. Conformance inspection

Examination or Test	Conformance Criteria		Requirement Paragraph	Inspection Method	Defect Classification
	Sample	Acc/Rej			
Examination for defects					
Cup, primer	Table IV	0/1	3.2/3.10	Table IV	Table IV
Button, primer		0/1			
Cup, support, primer		0/1			
Primer, electric		0/1			
Insulation Strength	100%	see 1/	3.3	4.4.1	Major

Electrical resistance	100%	see 2/	3.4	4.4.2	Major
Pellet weight	100%	see 3/	3.5	4.4.3	Critical
Electric primer sensitivity	800	see 4/	3.6	4.4.4	Major
Electric primer time	50	see 5/	3.7	4.4.5	Critical
Action time -65°F	50	see 6/	3.8	4.4.6	Critical
Function and casualty	300	Table VII	3.9	4.4.7	Major

Notes:

1/ A cup-insulator button assembly which fails to comply with the insulation strength requirement shall be rejected.

2/ A primer which fails to comply with the applicable electrical resistance requirement shall be rejected.

3/ A primer which fails to comply with the applicable minimum dry weight requirement shall be rejected.

4/ If the average firing voltage (V) plus three standard deviations (3σ) exceeds 160 volts, the lot shall be rejected or the first article sample will fail. For conformance inspection, the lot shall be subject to retest. If on retest, V + 3σ exceeds 160 volts the lot shall be rejected. If in either the first test or the retest, a primer fails to fire at 160 volts, the lot shall be rejected (see 6.8).

5/ If the sample average primer time plus four standard deviations exceeds 300 microseconds or if an individual primer time of the test sample exceeds 300 microseconds, the lot shall be rejected or the first article sample will fail (see 6.8).

6/ If the sample average action time plus four standard deviations exceeds 3.5 milliseconds, or an individual primer action time of the test sample exceeds 3.5 milliseconds, the lot shall be rejected or the first article sample will fail (see 6.8).

TABLE IV. Examination for defects

Primer Cup, Dwg. 7548057				
Classification	Examination or Test	Conformance Criteria	Requirement Paragraph	Inspection Method 1/
Critical	None defined	N/A	N/A	N/A
Major				
101	Cracks, split, or lamination	Level IV	3.10	Visual
102	Dent, ragged edge (V or U)	Level IV	3.10	Visual
103	Wire-like edge, or slivers lodged in or attached to cup	Level IV	3.10	Visual
104	Foreign matter, stain or corrosion	Level IV	3.10	Visual
Minor				
201	Height	Level II	3.2	Gage
202	Outside diameter	Level II	3.2	Gage
203	Scratch or nick	Level II	3.10	Visual
204	Evidence of poor workmanship	Level II	3.10	Visual
Primer Button, Dwg. 7548058				
Classification	Examination or Test	Conformance Criteria	Requirement Paragraph	Inspection Method 1/
Critical	None defined	N/A	N/A	N/A
Major				
101	Improperly formed	Level IV	3.2	Visual
102	Cracks, split, or lamination	Level IV	3.10	Visual

103	Dent	Level IV	3.10	Visual
104	Scratch or nick	Level IV	3.10	Visual
105	Foreign matter, stain or corrosion	Level IV	3.10	Visual
Minor				
201	Overall height	Level II	3.2	Gage
202	Outside diameter	Level II	3.2	Gage
203	Web thickness	Level II	3.2	Gage
204	Evidence of poor workmanship	Level II	3.10	Visual

Primer Support Cup, Dwg. 7548108

Classification	Examination or Test	Conformance Criteria	Requirement Paragraph	Inspection Method 1/
Critical	None defined	N/A	N/A	N/A
Major				
101	Ragged edge (V or U)	Level IV	3.10	Visual
102	Flash hole missing or obstructed	Level IV	3.2/3.10	Visual
103		Level IV	3.2	Visual
	Edge crooked or eared			
104		Level IV	3.10	Visual
	Burr at flash hole			
105		Level IV	3.10	Visual
	Foreign matter, stain or corrosion			
Minor				
201	Height	Level II	3.2	Gage
202	Outside diameter	Level II	3.2	Gage
203	Crack, split, or lamination	Level II	3.10	Visual
204	Scratch, nick, or dent	Level II	3.10	Visual
205	Evidence of poor workmanship	Level II	3.10	Visual

TABLE IV. Examination for defects – Continued

Electric Primer, M52A3B1, Dwg. 7548066				
Classification	Examination or Test	Conformance Criteria	Requirement Paragraph	Inspection Method 1/, 3/
Critical				
1	Missing, inverted, or insecure support cup	100%	3.2	AAIE
2		100%	3.10	AAIE
3	Oil or grease on electric primer	100%	3.2	AAIE
	Inverted button			
Major				
101	Mixed types	Level IV	3.2	Visual
102	Crushed or mutilated	Level IV	3.10	Visual
103	Missing, misplaced, or protruding insulator	Level IV	3.2	Visual
104		Level IV	3.2	Visual
105	No button or insulator covering button	Level IV	3.10	Visual
106	Presence of metal slivers on outside of primer or bridging the primer cup and button across insulator	Level IV	3.10	Visual
	Foreign matter other then oil or grease			
Minor				
201	Height	Level II	3.2	Gage
202	Diameter	Level II	3.2	Gage
203	Depth of button	Level II	3.2	Gage
204	Missing, slipped, or punctured disc	Level II	3.2	Visual
205		Level II	3.2	Visual

| 206 | Cocked support cup | Level II | 3.10 | Visual 2/ |
| 207 | Explosive composition on disc or support cup

Evidence of poor workmanship | Level II | 3.10 | Visual |

Notes:

1/ ORD-SIP-S314 shall apply in defining and evaluating cartridge visual defects. Defect classifications of ORD-SIP-S314 are revised as shown in Table V.

2/ Minute particles of explosive material are not cause for rejection.

3/ Automated acceptance inspection equipment (AAIE) shall be used to perform all critical defect inspections. AAIE shall be in accordance with MIL-A-70625 and approved by the government.

Table V. ORD-SIP-S314 Revisions

Component	Defect	SIP Classification	Revised Classification
Cup	Lamination	Minor	Permissible
Cup	Dent	Minor	Permissible
Cup	V or U	Minor	Permissible
Cup	Stain or corrosion	Minor	Permissible
Cup	Scratch or nick	Major	Minor
Button	Dent	Minor	Major
Button	Stain or Corrosion	Minor	Permissible
Button	Scratch or nick	Minor	Major
Support Cup	Stain or corrosion	Minor	Permissible

Support Cup	V or U	Minor	Permissible
Support Cup	Flash hole missing or obstructed	Minor	Major
Support Cup	Edge crooked or eared	Minor	Major
Primer Assembly	Inverted button	Major	Critical
Primer Assembly	Missing, misplaced, or protruding insulator	Minor	Major
Primer Assembly	Foreign matter other than oil or grease	Minor	Major

4.4 Method of inspection.

4.4.1 <u>Insulation strength</u>. The insulation strength of the cup-insulator-button assembly shall be determined applying the specified current, voltage, and timing.

4.4.2 <u>Electrical resistance</u>. Each primer shall be tested for the electrical resistance limits of 3.4. The inspection equipment shall limit the current and voltage to 1.4 milliamperes and 7.5 volts.

4.4.3 <u>Pellet weight</u>.   The minimum total dry weight of the primer mix shall be determined by measuring the recompressed bridge thickness of the primer. Bridge thickness is defined as the dimension between the center of the outside face of the primer button and the disc, or between the center of the outside face of the primer button and the exposed face of the primer support cup. The dry primer mix in the primer assembly shall be recompressed prior to measuring the bridge thickness using force of approximately 100 pounds in excess of the pressure required to move the cup support with respect to the primer cup. The minimum bridge thickness dimension used to assure minimum total dry weight of the primer mix and the recompression force shall be established by the supplier and approved by the procuring activity.

4.4.4 <u>Electric primer sensitivity</u>. The method of test shall be as specified in SCATP-20. The primer firing device shall be adjusted for a 10 microsecond discharge time from a 2 microfarad condenser. The electrical sensitivity limit of the sample primers shall be determined using the specified test method and procedure.

4.4.5 <u>Electric primer time</u>.   The method of test shall be as specified in SCATP-20. The test equipment shall be adjusted to comply with the specified current and voltage requirements.

4.4.6 <u>Action time</u>. The method of test shall be as specified in SCATP-20. The test sample primers shall be assembled as Cartridge, 20MM, Target Practice, M55A2, conforming to drawing 7258817, or Target Practice, PGU-27, conforming to drawing 1575AS300, and inspected in accordance with 4.4.7. The test cartridges shall be conditioned at -65°F for a period of not less than four hours after the conditioning chamber has stabilized at -65°F $\pm$ 5°F. Timing

for the conditioning period shall start after the chamber has stabilized following the sample being placed in the conditioning chamber.

4.4.7 Function and casualty. The method of test shall be as specified in SCATP-20. The test sample primers shall be inserted into cases that have been previously inspected for depth and diameter of primer pocket and length to shoulder basic diameter. The sample primed cases shall be assembled as Cartridges, 20MM, Target Practice, M55A2, conforming to drawing 7258817; or Target Practice, PGU-27, conforming to drawing 1575AS300. Prior to firing, the test cartridges shall be inspected for depth of primer seating, primer crimp missing, loose primer, metal slivers on case head of primer, profile and alignment max., and presence of foreign matter on primer button. The sample test cartridges at ambient temperature shall be fired in bursts of 50 rounds in the quantities and weapons as specified in Table VI below. The gun barrels shall be at ambient temperature at the beginning of each burst.

TABLE VI. Function and casualty testing

Accepted primers will be used in	M61 Weapon (5500 SPM, min)	M39 Weapon (1400 SPM, min)
20mm PGU Ammunition:	300	
All other 20mm ammunition:		300 1/
Primer First Article Test:		1100 1/

1/ Customers, through the procuring activity, may substitute the M61 Weapon for acceptance of primers that will be used to fulfill their orders.

TABLE VII. Casualties

Defect Description	Accept	Reject	Cumulative Reject	Classification
Missing button	0	1	N/A	Major
Misfire	0	1	N/A	Major
Primer leak 2/	3	9	9	Minor
Blown primer 1/	0	1	N/A	Major

Notes:

1/ The sample cartridge case shall be classed defective only if it is evident by visual inspection that both the primer pocket and case head are enlarged and deformed.

2/ If during conformance inspection, more then three but less then nine primers show evidence of leakage, a second sample of double the number specified by Table III (Production Lot Only)

shall be tested. If, in the accumulated samples, a button is missing, a misfire occurs, or nine or more primers show evidence of leakage, the lot shall be rejected. Misfiring test cartridges test shall be retested in the equipment specified in 4.4.6. Any misfires during retest shall be classified as to cause of failure and recorded as misfires in the test record.

5. PACKAGING

5.1 <u>Packaging</u>. For acquisition purposes, the packaging requirements shall be as specified in the contract or order (see 6.2). When packaging of materiel is to be performed by DoD or in-house contractor personnel, these personnel need to contact the responsible packaging activity to ascertain packaging requirements. Packaging requirements are maintained by the Inventory Control Point's packaging activities within the Military Service or Defense Agency, or within the military service's system commands. Packaging data retrieval is available from the managing Military Department's or Defense Agency's automated packaging files, CD-ROM products, or by contacting the responsible packaging activity.

6. NOTES

(This section contains information of a general or explanatory nature that may be helpful, but is not mandatory.)

6.1 <u>Intended use</u>. This item is military unique, and these primers are intended for ammunition to be used in U.S Army, Navy, Marines and Air Force 20mm automatic gun systems that have been designed for firing cartridges having the M52 type primer configuration.

6.2 <u>Acquisition requirements</u>. Acquisition documents should specify the following:

a. Title, number and date of this specification and all reference documentation cited in this specification (see 2.2.1).

b. Requirements for submission of first article: A first article sample, either in part or complete (Table II), may be required for the commencement of production after the award of a new contract, a change in production venue, a process change for any part or subassembly, or after a production stoppage in excess of 90 days as directed by the government contracting officer.

c. Requirement for submission of inspection equipment designs and manufacturing process.

d. Requirement and provisions for submission of test data as required.

e. Provisions for the inclusion of MIL-STD-1168, Ammunition Data Cards on DD form 1423, Contract Data Requirement List.

f. Provisions for critical characteristic controls.

g. Serialization requirements, if applicable.

h. Critical inspection equipment requirement.

i. Quality Conformance inspection, other than specified in Section 4 of this specification.

j. Applicable National Stock Number.

k. Lists of drawings, publications and specifications, showing applicable revision dates.

l. Certificate of conformance for each lot or shipment of product, if applicable.

m. Place of inspection, if not at place of manufacture.

n. Government Furnished Material or Equipment

o. Packaging, Packing, Marking and Unitization: For packaging and marking of inner containers, reference ARDEC drawings 9329662 and 9329663. For packing and marking of outer wire bound box, reference ARDEC Drawing 9329664. For unitization, reference DACs drawing 18-48-4116/159A-20PA1002.

6.3 <u>Automatic acceptance inspection equipment (AAIE)</u>. Provision concerning the AAIE used to verify the requirements of this specification should be specified in the contract if applicable.

6.4 <u>Submission of inspection equipment designs for approval</u>. Submit copies of designs as required to: Commander, US Army ARDEC, Attn: AMSRD-AAR-QEM-F, Picatinny, NJ 07806-5000. This address will be specified on the Contract Data Requirements List, DD Form 1423 in the contract.

6.5 <u>Firing tests</u>. In order to minimize inspection costs, the firing tests will be performed after the sample has been provisionally accepted for all other requirements. Additional cartridges may be required by the test facility (see 4.2). Tests may be performed concurrently on the sample cartridge provided that the test results are not affected by this procedure to minimize testing costs.

6.6 <u>Test validity</u>. If for any reason the test activity considers that the test conditions have detrimentally affected the test results, the test activity may request the Government to declare the test invalid and authorize a new test.

6.7 <u>Intermediate point inspection</u>. The classification of defects identifies the defect characteristics for acceptance inspection. It may be necessary to modify the sequence of inspection stations to best suit the manufacturing process. Inspection for defect characteristics which will be hidden or altered by subsequent processing operations (including unrelated operations), should be scheduled to prevent premature acceptance which could be detrimental to the attainment of optimum product quality of the end item.

6.8 <u>Standard Deviation</u>. Standard deviation (σ) should be calculated from the following formula or other approved formula:

$$\sigma = \sqrt{\frac{\sum\left(X_i - \overline{X}\right)^2}{n-1}}$$

where: = Each individual value X_i

$\overline{X}$ = Sample arithmetic mean

n = Sample size

6.9 <u>Malfunction or casualty not covered by this specification</u>. If a lot is suspended due to a malfunction or casualty not covered by this specification, the lot should be referred to the contracting officer.

6.10 <u>Changes from previous issues.</u> Asterisks and marginal notes are not used in this revision to identify changes with respect to the previous issue due to the extensiveness of the changes.

6.11 Critical defects.

6.11.1 <u>Hangfire</u>. A hangfire occurs when action time of a round is sufficiently long that the bolt unlocks before the projectile leaves the muzzle of the barrel. This results in unrestrained combustion and possible firing of the projectile out of battery. This could cause massive damage to the weapon system and weapon stoppage (see 6.11.2). In addition, in an aircraft application, due to the weapon's proximity to the user a hangfire will also present a safety hazard due to fragmentation and shrapnel from the weapon and the round.

6.11.2 <u>Weapon stoppage</u>. Due to the stand alone nature of the Land-based Phalanx Weapon System, when an ammunition defect prevents the weapon from functioning, this is considered a Mission Failure.

TABLE V. Critical defect justifications

Critical Defect	Justification
Missing, inverted, or insecure support cup	This could cause degradation of the primer pellet resulting in a low weight primer pellet and a hangfire.
Oil or grease on electric primer	Oil or grease on the primer has been demonstrated to lower the output of the primer to the extent that a hangfire results.

Inverted button	An inverted primer button would cause a low weight primer pellet and a hangfire.
Action time	Long enough action time will result in a hangfire.
Electric primer time	Long primer times could affect primer output or action time which could result in a hangfire.
Pellet weight	A low weight primer pellet is a demonstrated cause of hangfires.

6.12 Subject term (key words) listing.

 20 millimeter

Electric Primers

Custodian: Army-AR

Preparing activity: Army-AR   (Project 1305-2006-001)

NOTE: The activities listed above were interested in this document as of the date of this document. Since organizations and responsibilities can change, you should verify the currency of the information above using the ASSIST Online database at http://assist.daps.dla.mil

Appendix D: Health and Safety Documents

APPROVAL AND REVIEW				
SOP NUMBER **10090-03**		**CODE** **478300D**		**EXPIRATION DATE (Month/year)**
TITLE Pressing of energetic material				
LOCATION **Building 10090**				
OTHER APPLICABLE SOP'S, PARTS, ETC. **IDP 4062 Section 1 NAVSEA OP-5, NAWSCLINST 5100.4,**				

PREPARED BY	CODE	DATE	CONCURRED BY	CODE
Cynthia Lovern	**478300D**		**Susie Johnson**	**470000D**
CONCURRED BY	**CODE**	**DATE**	**REVIEWED BY (Safety Program Office)**	**CODE**
Gabe Soto	**478300D**		**Roger Zurn**	**N43NW**
CONCURRED BY	**CODE**	**DATE**	**APPROVED BY (Department head)**	**CODE**
Phil Dixon	**478000D**		**Barry Hand**	**470000D**

REMARKS:

SOP requires continuous review to ensure that it is current. Team Leads (TL's) are responsible for ensuring that the SOP is continuously reviewed and that minor changes have been incorporated into the current SOP. If major changes are needed, the TL shall ensure that the SOP is revised and reviewed and approved by all elements involved in the SOP development. TL's and Team Members (TM's) shall sign the Process Supervisor's Statement or Worker's Statement yearly to indicate their continuous review of the SOP.

1. SOP Validated by _____ Date _____.

   by _____ Date _____.

Executive summary of work covered under this SOP:

This SOP details the procedure and safety required to press energetic and experimental material to produce pellets, boosters, leads, and detonators. Some of these materials are extremely sensitive and could react to any applied force making a reaction an expected event. If a reaction does occur, it will be considered a result of the process and not an explosive incident. The operation will stop and all equipment will be checked for damage. The supervisor will be notified and a decision will be made as to whether the particular material will continue to be pressed. This SOP describes the process with all computer-numerically-controlled (CNC) presses and the Dead Load Press.

TABLE OF CONTENTS

WORKER AND SUPERVISOR STATEMENTS

Process Supervisor (Team Lead (TL))

I have read and understand this SOP. To the best of my knowledge, the processing described within this SOP can be done in a safe, healthful and environmentally sound manner. I have made sure all persons assigned to this process are qualified, have read and understand the requirements of this SOP, and have signed the worker's statement for this process. I will ensure the SOP has current procedures. If a major change to the SOP is necessary, I will ensure that the process is stopped until the SOP is revised and approved. If unexpected safety, health, or environmental hazards are found, I will make sure the process is stopped until hazards have been eliminated.

Worker (Team Member (TM))

I have read this SOP and I have received adequate training to perform the process according to the SOP. I will follow the SOP unless I identify a hazard not addressed in it or encounter an operation I cannot perform according to the SOP. If that occurs, I will stop the process and notify my immediate supervisor of the problem.

QUALIFICATION AND CERTIFICATION STATEMENTS

Qual/ Cert Level	FO Training Course Date	BEST Course Date	NAME	Code	Date	Signature
TL	4/02	9/97	Joel Huddleston	478300D		
TL	3/06	1/99	Michael Aramanda	478300D		
TL	4/02	2/02	Rod Robbs	478300D		
TL	3/06	10/01	Michael Woodward	478300D		
TL		1/07	Cynthia Lovern	478300D		
TM		8/06	John Carroll	478300D		
TM		04/07	Anthony Joyce	478300D		
TM		03/07	Robert Burrows	478300D		
TM		08/07	Paul Winkel	478300D		

TL – Team Leader, TM – Team Member

HAZARD CONTROL BRIEFING

Sample Briefing Topics:

1.   Are personnel, building, and equipment ready for safe operation?

2.   Are weather conditions acceptable?

3.   Are safety features operational?

4.   Is proper PPE is being worn?

5.   Have conductive shoes been checked (each day prior to working with sensitive explosives)?

6.   Has a safe man been established?

7.   Have hazards associated with specific explosive components involved in current operations and mitigation controls been discussed?

8. Are Explosive limits being maintained?

(a) Type 1. This type is all-inclusive. It addresses the general hazards associated with energetic material operations and describes the basic hazards that workers encounter while conducting routine tasks associated with energetic operations. These routine tasks include, but are not limited to, handling, transportation, and storage of energetic materials. The HCB must address the hazards and the methods to control them. Personnel must successfully complete this type of hazard control briefing before being assigned the duties of an explosive worker or supervisor. Basics of Naval Explosives Hazard Control (AMMO-18), and General Explosives Indoctrination Course (or equivalent), fulfill this requirement (see reference (f)).

(b) Type 2. This type of HCB will address those hazards that are particular to each operation. Type 2 HCBs will focus on the operation and work area as contained in the operating procedures, and will address the hazards and controls for each operation as discussed in the ORM. These briefings can be conducted at the job site before the operation or during regular safety meetings. However, a Type 2 HCB will be conducted before each and every operation that presents an unusually high risk. Those operations that present unusually high risks will be identified by the ESO during the initial approval review. Type 2 HCBs are required at least monthly or when SOPs are changed. In addition, a simplified Type 2 HCB will be given to all visitors before they are allowed to enter any operating or hazard area. Training is required monthly for those SOPs in continuous use. For less frequently used SOPs, training must be provided before their use. Particular attention is needed when changes are made to SOPs or when SOPs are reinstated after periods of inactivity.

(2)   Documentation of HCBs is required. Maintain records for each briefing conducted. Documentation should include the names of attendees, dates, and title of the SOP or subjects presented. Training records documenting formal classroom training for Type 1 HCB will be maintained by each competency. The Safety and Occupational Health Division, Code 844000D, maintains a limited data base for training completed.

NAME	JANUARY	FEBRUARY	MARCH	APRIL	MAY	JUNE	JULY	AUGUST	SEPTEMBER	OCTOBER	NOVEMBER	DECEMBER
Joel Huddleston												
Rod Robbs												
Michael Woodward												
Michael Aramanda												
John Carroll												
Cynthia Lovern												
Anthony Joyce												
Robert Burrows												
Paul Winkel												

HAZARD CONTROL BRIEFING

I certify that I have had a hazard control briefing on SOP# **10090-03**
SOP Title: **Pressing of Energetic Materials.** The hazard control briefing will be given at least once prior to the use of this SOP. The briefing shall be given each time a new team member (TM) or team Lead (TL) is assigned to this SOP. The briefing shall be given if this procedure has not been performed within the last 30 days.   Please print your name and initial the month this briefing was attended.

HAZARD ANALYSIS/EVALUATION

HAZARD TYPE	HAZARD DESCRIPTION	PRECAUTIONS
Explosives	Explosion, fire, or both	Follow ordnance handling procedures, and any additional safety precautions as described in the HCB
Propellants	Explosion, fire, or both	Follow ordnance handling procedures, and any additional safety precautions as described in the HCB
Pyrotechnics	Burns	Follow ordnance handling procedures, and any additional safety precautions as described in the HCB
Heat, flame	Burning propellant caused by normal execution of test.	Non-essential personnel will be outside the controlled area access.
HERO	Premature Initiation	No radio transmissions within 50 feet of HERO sensitive elements.

ENERGETIC MATERIAL LIMITS

SITE (Room #/Area)	PERSONNEL		HAZARDOUS MATERIALS				
	Operator	Transient	TYPE		AMOUNT		
			CLASS	DIV.	QTY.	UNIT	DESCRIPTION/COMMENTS
Bldg. 10090 104	2	2	1.1, 1.4		60	LB	Shipping Room
							Long Room – General disassembly and assembly

105	2	2	1.1, 1.4		10	LB	
121	1	1	1.1, 1.3, 1.4		2	LB	Pressing Room
125	1	1	1.1, 1.3, 1.4		1	LB	Firing Room
127	1	2	1.1, 1.3		0.25	LB	MEMS Lab
129	1	1	1.1, 1.3, 1.4		1	LB	Small Room – General disassembly and assembly
Bldg. 10100							
1	2	1	1.1, 1.3, 1.4		50	LB	General disassembly and assembly
Bldg. 10060							
101	2	2	1.1		1000	LB	Long Room- General disassembly and assembly
105	2	1	1.1		125	LB	Pressing Room
107	2	1	1.1		125	LB	Small Room- General disassembly and assembly

NOTES:

1. NONE OF THE PERSONNEL ENERGETIC MATERIAL LIMITS EXCEED SITE APPROVAL LIMITS.

2. NONE OF THE PERSONNEL ENERGETIC MATERIAL LIMITS EXCEED SITE POSTED LIMITS.

EMERGENCY RESPONSE and CONTINGENCY PLANS

A. In case of emergency:

(1) Use nearest fire alarm box or telephone 911. When using a cell phone, dial 939-3323. Know the location of the nearest alarm box and nearest telephone in the area. When using a telephone, REPORT the five digit BLDG number and where you will meet emergency personnel.

(2) In case of evacuation, proceed to nearest exit and gather at designated area(s).

(3) All personnel handling energetic and/or hazardous materials will be encouraged to maintain certification in CPR and First Aid.

B. See posted evacuation plan.

C. Emergency Phone Numbers

SERVICE	DAY	NIGHT
FIRE, AMBULANCE, POLICE	911	911
MAINTENANCE/UTILITY EMERGENCY: Lyndon Martinsen Michael Heseman	939-9402 939-9402	446-3356 608-3547 375-3471 382-6965
NOTIFY AS SOON AS TIME PERMITS		
DEPARTMENT OFFICE:		
ASSOCIATE FOR ENERGETICS – RANDY COPE	939-7759 382-8810 (CELL)	375-3270

OR DIRECTOR FOR OPERATIONS & SAFETY – BARRY HAND	939-7121 382-6830 (CELL)	377-3620
OR SAFETY AND OPERATIONS OFFICER – SUSIE JOHNSON	939-7465 382-6847 (CELL)	375-4791 677-7108 (CELL)
SAFETY OFFICE STAN AUSTIN BILLMCCRACKEN ROGER ZURN INDUSTRIAL SAFETY TOM REA	 939-0967 939-2040 939-3167 939-7753	 375-8998 499-1425 917-3322 382-8237

FIRE BILL

FOR

EXPLOSIVES AREAS

BUILDING NO.'s 10060, 10090, 10100

1. TURN IN ALARM BY: A. TELEPHONE   911

B. PULLING NEAREST ALARM BOX

C. ENGAGING GRINNEL SYSTEM

2. EVACUATE ALL PERSONNEL TO THE FIRE DIVISION MEETING PLACE AT THE FOLLOWING LOCATION:

_____**2nd and J**_____

3. NOTIFY PERSONS IN NEARBY BUILDINGS OF IMPENDING FIRE DANGER

4. ESTABLISH ROAD BLOCKS AT:

1: _____

2: _____

3: _____

4: _____

5: _____

REVIEWED BY: _____ **DATE:** _____

REVIEWED BY: _____ **DATE:** _____

REVIEWED BY: _____ **DATE:** _____

REVIEWED BY: _____ **DATE:** _____

NOTES

This SOP details the procedure and safety required to press energetic and experimental material to produce pellets, boosters, leads, and detonators. Some of these materials are extremely sensitive and could react to any applied force making a reaction an expected event. This SOP describes the process with all computer-numerically-controlled (CNC) presses and the Dead Load Press. This procedure shall be used in compliance with the Building 10090 General Area Instruction Building oR Site Diagrams:

  a. Energetic Material and Personnel Limits: Each facility shall have the personnel and explosives limits (PELs) posted at each entrance.
  b. Building site diagrams and location of safety related items shall be posted at each emergency exit.
  c. Emergency exits are posted.
  d. PROCESS FLOW DIAGRAMS ARE NOT APPLICABLE

OPERATIONS EQUIPMENT LIST:

ITEM	DESCRIPTION
01	Computer and CNC operating software/CNC Presses or Dead Load Press
02	Explosive Proof Scale
03	Scoops, spatulas, and containers of a non-sparking material

ITEM	DESCRIPTION
04	Die set and pressing fixture
05	Grounded workbench/table

SAFETY EQUIPMENT LIST:

ITEM	DESCRIPTION
01	Steel Toe Safety Shoes (conductive)
02	Flame Resistant Coveralls
03	Safety Glasses with Side Shields
04	Hearing Protection
05	Pliers
06	Respirator

OCCUPATIONAL HEALTH HAZARDS CONTROLS:

e. All operators have been briefed on all the hazardous materials used in the facilities and the availability of MSDSs for these materials.

f. MSDSs are available in the Branch Office and control rooms, reachable within five minutes or immediately by telephone.

SECURITY STATEMENT

1. ALL CLASSIFIED MATERIAL WILL BE HANDLED IN ACCORDANCE WITH OPNAV 5510.36

2. ALL ENERGETIC MATERIAL WILL BE HANDLED IN ACCORDANCE WITH OPNAV OP 5 AND NAWCWPNS IDP 4062.

OPERATING PROCEDURES AND INSTRUCTIONS

OPERATION	DETAILS OF OPERATION	SAFETY FACTORS/LIMITS
1. Observe electrostatic metering (field mills) for the area.	1A. When the electric field reaches or exceeds 2000 volts per meter due to an approaching electrical storm.	1a. All ordnance operations shall be shutdown and personnel evacuated to a safe location. Command approval must be obtained in order to continue operations.
	1B. When the electric field reaches or exceeds 2000 volts per meter due to a wind/dust storm, the hazard is static electricity.	1b. Shutdown of operations shall be determined on a case-by-case basis by the personnel performing the work. Personnel to consider the electrostatic sensitivity of the material or item being worked on and where the work is being performed.

(1)Outside work on all but the most electro-statically insensitive items (e.g., un-fuzed bomb) shall cease.

(2) Inside work with electro-statically sensitive items or materials (e.g., hot wire electro-explosive devices and black powder) shall cease.

1c. Inside work with less |

		sensitive items or materials (e.g., secondary explosives and propellants with high electrostatic initiation thresholds) may continue.
		1d. Always work with the minimum amount of energetic material possible.
2. Check personnel, building, and area for safe operation.	2A. Review information applicable to the operation.	2a. Operations shall not proceed until it is safe to do so (i.e., warning flags are up, rotating beacon is on, personnel are wearing PPE, building and area safety features are operational, a safe man has been established, weather conditions are acceptable, Hazard Control Briefing has been conducted, etc.) All requirements of Part II of this SOP shall be met before operations begin. Safety glasses must be worn at all times. Operator must be grounded through conductive shoes and conductive floor when handling electrically initiated explosive components.
2. Check personnel, building and area for safe operation, continued	2B. The supervisor of the operation shall verify that the building(s) are safe for the operation (s). 2C. The supervisor of the operation shall verify that no personnel other than those involved in the operation are in the area.	Flame-resistant coveralls or lab coats are required at all times. Cotton clothing is required under the coveralls or lab coat. Equipment must be grounded. Observe all

		precautions associated with energetic material hazards (electrostatic, impact, friction, flammability, toxicity, explosion, electrical, and chemical incompatibility)
3. Preparation	3A. Check that the work surfaces are clean, dry and grounded.	3a. Ensure work area is clear and that all necessary or special tools are available to complete the procedure. Prior to conducting any procedure, inspect munition/component for damage and ensure manufactured safeties (if applicable) are in place. If the item is damaged beyond the scope of this SOP (e.g., leaking, exuding, corroding, etc…) or cannot be verified to have passed electronic quality assurance, coordinate transfer to the magazines or EOD.
	3B. Clean and assemble tooling as required.	3b. Visually inspect and clean all tooling.
	3C1. **Dead Load Press:** -Set the press to the desired force by adding weight to the beam. 3C2. **H5KS Press:** -Attach laptop if necessary, turn on power breaker, launch Navigator software.	3c. Density and weight calculations will be done before the process begins.

3. Preparation, continued		
	3C3. **15 Ton Tinius Olsen Press:**	
	-Apply 5psi of air pressure to inlet of press housing. Turn on power breaker in control room. Boot computer and launch Navigator software.	
	-For remote operations, place camera in position to watch press and die. Launch viewing software on computer.	
	3D. For CNC machines, program the appropriate pressing settings for fixture and material in CNC software as per test plan.	3d. Refer to CNC software documentation as necessary. Adjust force and deflection to settings specified in test plan.
	3E. Adjust the scale to zero.	
	3F. Fill the tooling with inert material. Move press head into desired position, zero the load cell and position sensors.	
	3G. Press the inert material to verify proper function of tools and loaded pressing procedure (software).	3g. Inspect the finished product tooling for gouging or scratches. The die set must be cleaned after every pressing.
		-WARNING-
4. Loading and Pressing	4A. Weigh the powder.	4a.-USE CALIBRATED BEAM SCALES OR EXPLOSIVE PROOF ELECTRONIC SCALES.

		-HANDLE ONLY THE MINIMUM AMOUNT POSSIBLE. -USE ONLY TOOLS OF A NON-SPARKING MATERIAL. -PERFORM EVERTHING BEHIND A SHIELD.
4. Loading and Pressing, continued	4B. Transfer the weighed charge to the die.	4b. Perform behind a safety shield.
	4C. Insert ram in die (using pliers if deemed appropriate) and place die in press. If remote operation is specified, proceed to control room, otherwise, run press from behind shield as per test plan.	4c. Inserting the ram into the die will be done behind a shield. Remote operation will be conducted when material exceeds 10 grams or if specified in test plan.
	4D. **Dead Load Press:** -Release pressure and raise press head clear of the die.	4d. The press head must be completely clear of the die before opening door to the press.
	4E. **CNC Machines:** -Verify that the press head has returned to the zero position after pressing by sensor reading and visual inspection.	
	4F. Remove the die set from the press (using pliers if	4f. Removing/inserting the

	deemed appropriate). Wipe down ram between presses. As required, add more powder to the die for multiple increments or install the closure disc and place the die back in the press to press or crimp.	ram will be done behind a safety shield. Pressing and crimping will be performed behind a safety shield or done remotely if applicable.
	4G. Remove the finished unit from the loading sleeve.	4g. Perform behind safety shield. Use arbor press as necessary to remove unit from die.
	4H. Clean the die set.	4h. The die set must be wiped clean after every pressing to remove excess or spilled powder.
	4I. Document applicable data on product sheets as required, or use the automatic reporting of the pressing software.	**-NOTE-** Keep cleaner solvents (ie Acetone) out of the pressing room while powder is open to prevent contamination.
5. Completion of Loading	4J. Repeat steps A-I as required until operation is complete. 5A. **CNC Machines:** -Shutdown and safe machine per operating manual. 5B. **Dead Load Press:** -Remove weight from the beam. 5C. Clean work area and tools. 5D. Dispose of all explosive scrap as hazardous waste.	5c. Remove explosive materials from the pressing room or seal in secure container prior to use of cleaner solvents to prevent contamination.

6. Clean Up	6A. Coordinate transfer of explosive/explosive components to the magazine or EOD for further disposition.	**NOTE** Clean all tools and work surfaces that have come in contact with explosives with the required neutralizing agent as specified in the work request/MSDS. All EHW shall be processed in accordance with Addendum A.
	6B. Coordinate transfer of explosive samples to the chemical lab for analysis, or to customer for further disposition.	
	6C. Clean and return all tools to appropriate storage and report any damaged/broken tools or equipment.	
7. Emergency Procedures	7.A In case of accident or injury, obtain emergency help if needed, then:	
	1. Cease all operations	
	2. Notify Area Supervisor	
	3. Notify Division Safety Representative	
	4. Send personnel to emergency meeting place, if necessary.	
8. Emergency Shutdown	8A. In case of emergency or the need to shutdown quickly occurs:	Joel Huddleston: 939-0854
		382-8804cell
	1. Stop work.	Susie Johnson: 939-7465
	2. Leave building and lock the doors.	382-6847cell

GENERAL AREA INSTRUCTION

SOP: 10090-03	BUILDING: 10060,10090,10091,10095 (DROP TOWER) and all magazines	CODE: 478300D
Describe operations, details of operations, safety factors/limitations, number of personnel, and amounts of energetic material for each operation, etc. Provide step-by-step procedures and allowable scale-up and/or variation limits, where applicable and appropriate.		
OPERATION	**DETAILS OF OPERATION**	**SAFETY FACTORS/LIMITS**
1. General Requirements	1A. The red warning flag will be put up anytime personnel and explosives are in the area. Flashing red light is o be used is a test is imminent or in progress or drying of explosive is in progress. 1B. Conductive shoes shall be worn in authorized spaces only. Personnel working with primary explosives/sensitive EED'S must make daily shoe conductivity checks. 1C. Flame resistant coverall shall be worn by all personnel working with or handling hazardous materials/explosives or who are close enough to be burned if the materials ignite. 1D. Safety glasses with permanently attached side shields shall be worn when working with hazardous or energetic materials, while operating machine tools, or performing any task in which an eye hazard may exist. 1E. Hearing protection must be worn where a noise hazard exists. 1F. Whenever toxic vapors or dusts are present in a work area, suitable respiratory equipment shall be worn. 1G. Before starting a hazardous operation,	1c. Non-static producing clothing shall be worn under coveralls. 1g. A safe person is required to

	obtain a "Safe Man".	be present whenever explosives are being handled. The safe man must be notified of their responsibility. They must be located outside of the hazardous operation, but sufficiently close that they would be aware of and could provide immediate assistance.
	1H. Telephones shall be checked prior to commencing hazardous operations.	
	1I. All areas shall be kept clean and in good condition.	1h. Hazardous operations are not to be conducted when telephones are not functioning.
1. General Requirements (Continued…)	1J. Personnel and explosive limits for each operation and area shall be complied with at all times. 1K. All area warning signs and devices shall be checked frequently and repaired as needed. 1L. Modification of operations based on receipt of electrostatic or storm warning is required.	1i. Materials and gear shall be properly stowed at the end of each work day. Explosives scrap will be removed from the work spaces daily and placed in the "scrap" magazine. 1j. Personnel and explosive limits are posted inside each building.
	1M. All area personnel shall be responsible for complying with environmental regulations.	

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		1l. IAW OP-5, when electric field reaches or exceeds 2000 volts per meter, ordnance operations will cease and personnel will evacuate to a safe location.
2. Emergency Procedures	2A. Building/Site diagrams including the locations of telephones, fire extinguishers, eye washes, emergency showers, evacuations routes and emergency exits are posted inside and outside each building.	2a. Observe all emergency procedures as dictated by explosive safety policies and regulations.
	2B. ACCIDENT PROCEDURE: If an accident/injury occurs, call the Fire Department at 911. Apply first aid if necessary. Notify area supervisor.	
	2C. FIRE: DO NOT attempt to fight a fire involving energetic material. Evacuate to a safe location. Call 911. Send someone to meet the Fire Department at the designated meeting place. Notify area supervisor.	
	2D. SPILL: Clean up the spill if you have the knowledge and equipment to manage the spill. Otherwise call 911.	
3. Arms Ammunition and Explosives Security and Accountability.	3A. Any operations involving explosives that have special security concerns will be noted in an addendum to the particular SOP. Otherwise the SIM will ensure AA and E Accountability and security is followed.	

Hazard analysis/evaluation

SOP: 10090-03	**BUILDING: 10060,10090,10091,10095 (DROP TOWER) and all magazines**	**CODE: 478300D**
General Area Instruction/ Energetic Material Hazard Assessment		
HAZARD TYPE	**HAZARD DESCRIPTION**	**PRECAUTIONS**
1. Electrostatic	1A. Most materials in the plant are not considered a hazard; but since a few are, all materials will be considered electro-statically sensitive	1a. All equipment will be grounded. Excess explosives will be kept to a minimum and proper grounding techniques will be practiced whenever explosives are handled.
2. Impact and Friction	2A. Explosives and propellants can be ignited by impact and/or friction.	2a. All hazardous materials should be handled with caution. Avoid dropping explosive materials.
3. Flammability	3A. Energetic materials need outside source of heat to ignite. Ignition may be caused by heat or exposure to direct, focused sunlight.	3a. Keep all potential sources of heat production from energetic materials area.
4. Toxicity	4A. Dust from powders or solvents used for clean- up and some other ingredients may cause breathing and skin problems.	4a. Use proper ventilation and personal safety equipment, such as respirators and rubber gloves. Use knowledge of MSDS.
5. Chemical Incompatibility	5A. Exothermic reactions caused by chemical incompatibilities are possible.	5a. When bonding materials, care should be taken to identify that the adhesive is not chemically incompatible and that any heat generated by the curing process will not be sufficient to produce ignition.
6. Explosion	6A. Energetic materials are designed to initiate which will result in hazards from high pressure, shock, and fragments.	6a. Keep initiation sources (heat, shock, impact, friction, electrical current, and electrostatic discharge) isolated from energetic materials.

7. Electrical	7A. Energetic materials are subject to initiation by electric spark or current.	7a. All equipment will be grounded where possible. Bridge wire circuits will be shorted at all times.
8. Noise	8A. Initiation could produce a loud report that can damage hearing.	

GENERAL POLICY ADDENDUM FOR HANDLING, STORAGE, AND TRANSPORT OF ENERGETIC MATERIALS

SOP: 10090-03	**BUILDING: 10060,10090,10091,10095 (DROP TOWER) and all magazines**	**CODE: 478300D**

All Code 470000D China Lake Locations

Describe operations, details of operations, safety factors/limitations, number of personnel, and amounts of energetic material for each operation, etc. Provide step-by-step procedures and allowable scale-up and/or variation limits, where applicable and appropriate.

OPERATION	**DETAILS OF OPERATION**	**SAFETY FACTORS/LIMITATIONS**
1. Handling	1A. In conjunction with R & D operations within Code 470000D, most personnel are required to handle energetic materials in order to perform their assigned duties.	1a. (1) All personnel whose positions require them to handle energetic materials must take the Basics of Explosive Hazard Control Course or equivalent. (2) On-the-job training must be accomplished. (3) Hazard Briefs will be performed at least once a month, or in the case of inactive SOPs, before the start of the operation. (4) Current MSDSs and/or Hazardous Materials Traveler Sheets will be used to provide information on all materials

		handled.
	1B. Forklift equipment used for handling energetic materials will only be operated by trained personnel having valid industrial and ordnance forklift operator licenses.	1b. Trained forklift operators will follow the requirements of NAVSEA SW023-AH-WHM-0010.
2. Storage	2A. Raw materials, synthesized products, and new formulations are required to be placed in proper storage facilities.	2a. Only persons having key delegation of authority will be allowed to act as key custodians/sub-custodians. Keys must be properly stowed and storage facilities will be checked at day's end to assure they are secured.
		2b. Certified SIMS or their alternates having delegation of authority are responsible for keeping a proper log of items placed in or removed from storage and for meeting all ammunition and explosives management requirements (NAWSCLINST 8012.1).
		2c. Raw materials must be stored in buildings or refrigerated storage designated for each type of material; thus allowing segregation, temperature control, etc. necessary for maintaining acceptable operational materials.
		2d. Synthesized raw ingredients and new energetic formulations must be labeled with class/division and storage compatibility groups on each package before being placed in storage. Materials of unknown hazards must be segregated.
		2e. All stored items will have proper identifying information including NALC/DODIC or NAWS assigned numbers, explosive inventory cards, and condition code tags (All AA&E requirements must be followed).

		2f. Water wetted explosive containers will be checked for water content and weight of explosive per OP-5 at intervals of not less than one year or more than 2 years. Inspection records will be kept a minimum of 2 cycles.
		2g. When transferring raw ingredients from larger containers into smaller ones, these guidelines shall be followed:
		(1) If an ingredient is not considered to be an explosive, propellant, pyrotechnic or an otherwise hazardous material, drums can be opened and material withdrawn and transferred into smaller containers.
		(2) If the ingredient to be transferred is an ordnance material, the storage drum must be removed from the rest house or magazine and taken to an ordnance operating building for sample transfer.
		(3) All changes in weight, explosive inventory card entries, and condition code information must be tracked as required, making certain that each large and small receptacle is carefully labeled.
3. Transportation	3A. Energetic materials will only be transported by persons having a valid Explosive Safety Driver's license (including a medical card) and a demonstrated familiarity with NAVSEA OP-5, OP-2165, SW020-AF-ABK-010 and SW0023-AG-WHM-010	3a. ON-CENTER (1) A safe person must always be present during loading and downloading of energetic materials. Transporting ordnance within the confines of the Naval Air Weapons Station, China Lake, does not require additional personnel other than the permitted driver. (2) Appropriate placards must be

		displayed when transporting explosives.
		(3) Drivers must be aware of security measures required when transporting Risk Category I or II items (no police escort is required on-center).
		3b. OFF-CENTER
		(1) Off-center transportation must be according to CFR Title 49, Parts 171 to 179.
		2) DOT and UN regulations must be followed.

**GENERAL POLICY ADDENDUM FOR EXPLOSIVE HAZARDOUS WASTE (EHW)
AND SCRAP METAL DISPOSAL**

SOP: 10090-03	BUILDING: 10060,10090,10091,10095 (DROP TOWER) and all magazines	CODE: 478300D

All Code 470000D China Lake Locations

Describe operations, details of operations, safety factors/limitations, number of personnel, and amounts of energetic material for each operation, etc. Provide step-by-step procedures and allowable scale-up and/or variation limits, where applicable and appropriate.

OPERATION	DETAILS OF OPERATION	SAFETY FACTORS/LIMITATIONS
1. Background	1A. Explosive scrap shall be treated as hazardous waste. NAWS INSTRUCTION 5090.1 (series), ENVIRONMENTAL MANAGEMENT OF EXPLOSIVE HAZARDOUS WASTE AND ENERGETIC RANGE RESIDUE provides guidance of handling EHW and energetic range residue. Code 470000D designated Explosive Scrap magazines have been defined as Satellite Storage or Less Than 90 Day Storage Areas. Satellite Storage areas are restricted to a total solid weight of 458 pounds or less (NEW plus packaging) or 55 gallons of liquid hazardous waste. Hazardous Waste in a Satellite or Less Than 90 Day Storage area must have a Hazardous Waste Label affixed to the container. All safety, physical security, hazardous waste and inventory requirements for energetic materials remain in effect.	
2. Packaging	2A. Package scrap in a suitable closed container. 2B. Affix a Hazardous Waste Label (NAWCWD 6280/1) to the container.	

	Label shall have the following lines filled in: Code; Phone; Accumulation Start Date; Content, Contents, Composition. Check appropriate Physical State and Hazardous Properties block. Provide brief description of waste in block. Do not complete the waste code block. 2C. Affix Material Condition Code (MCC DD Form 1577) Unserviceable Condemned Tag-Material to waste. Fill out MCC and use Condition Code "H" for EHW. 3A. Stow container in appropriate scrap magazine. 3B. Add to scrap magazine inventory. 4A. Prepare manifest of waste to be treated in accordance with NAWSINST 5090.1 (series). Use NAWS 8027/2 (latest rev.) EXPLOSIVE ORDNANCE DISPOSAL TEAM JOB REQUEST. 4B. Contact Code 470000D to pick up scrap and take to EOD. 5A. Observe the same emergency procedures as dictated by explosive safety policies and regulations. 5B. FIRE: DO NOT attempt to fight a fire involving energetic materials. Evacuate to a safe location, notify the fire division, ext. 911, and send someone to the designated fire division meeting place. 5C. SPILL: Clean the spill up yourself if you have both the knowledge and equipment to manage the spill. Otherwise, call the fire division, ext. 911, and they will notify EOD to clean it up.	

	6A. Before starting the inspection, allow adequate time to elapse in order for items or fragments to cool. 6B. Examine metal parts and pieces. Look for un-reacted energetic material. -Examine crevices or minute openings to visually ascertain if they contain any un-reacted material. -Examine exposed threads used. 7A. If you can visually verify an item is empty, it can be sent for decontamination by the use of Citrus solvent, other solvents, or by Steam Cleaning. If there is no opening to verify an item is clean and decontaminated, have it treated by EOD. Accumulate these items as EHW until treatment is completed. 8A. Complete Generator section of NAWS 8027/6 for turn-in to the Range Residue Collection Facility. Authorized Code 47 Personnel with delegated authority must certify scrap safe for on-station transport/storage.	
3. Stowing		2a. Container must be compatible with the waste. Containers may be combustible or noncombustible. Cardboard, plastic, or metal containers are acceptable. Scrap is treated by EOD by open detonation. 3a. Insure materials are compatible with magazine contents and limits. 3b. Do not exceed the satellite scrap magazine's NEW limit or 458 pounds total waste including packaging. Do not

		exceed the Less than 90 day scrap magazine's NEW limit.
4. Treatment		4a. Explosive scrap shall be treated of monthly.
5. Emergency Procedures		
6. Inspection of expended metal test hardware and shell casings for on Station transfer and storage.		6a. Hot items or fragments can cause serious burns. 6b. Wear leather gloves to prevent cuts on hands from rough metal. Do not unscrew any plugs or covers to obtain access to interiors of test items. Un-reacted material in threads may be friction sensitive.
7. Contaminated/Cannot Verify for Contamination		
8. No Contamination Found		8a. Only personnel delegated IAW NAWSINST 8027.1 MANAGEMENT AND DISPOSITION OF MPPEH AND INERTING OPERATIONS, may sign this statement.

Appendix E: List of Nano Aluminum Powders and Properties

Sample	Source	Size	Vendor BET Surface Area m²/g	TGA	Active Al (Hydrolysis)	NAVAIR WD BET Surface Area m²/g	BET Size nm	TGA	Active Al (Hydrolysis)	ESD mJ	SEM	Time Aging	Loose Powder Density g/cc
	Nanotechnology	33 nm								1.25			
	Nanotechnology	50 nm	42.6	67.20%		41.4	46	67.9 %	66.50%	0.5	L.1		
M2210D	NovaCentrix	80 nm	25.1		87%			86.9 %		0.09	L.4		0.24
M2210D	NovaCentrix	80 nm	25.1		87%			74.0 %		15.6		5 yrs	0.24
M2210B	NovaCentrix	80 nm						79.0 %					
M2210B	NovaCentrix	80 nm						74.0 %	73.90%				
M2443	NovaCentrix	50 nm								2.5			
M2444	NovaCentrix	50 nm											
M2445	NovaCentrix	50 nm											
M2446	NovaCentrix	50 nm											
M2448	NovaCentrix	50 nm											
M2449	NovaCentrix	50 nm											
M2450	NovaCentrix	50 nm											
M2451	NovaCentrix	50 nm						61.4 %					
M2452	NovaCentrix	50 nm											
M2453	NovaCentrix	80 nm						81.1 %					
M2454	NovaCentrix	80 nm											
M2455	NovaCentrix	80 nm											
M2671	NovaCentrix	80 nm						77.6 %	81.7%				
M2697	NovaCentrix	80 nm	23.6		74%	23.6	86	70.9 %					0.37

NEF03-1	Technanogy	50 nm				44.83		66.7 %					
RF-A	LANL	50 nm						60.3 %			L.2		
	Technanogy	138 nm							85.40%				
ALEX	Argonide	150 nm							84.90%		L.3		
Al Flake	Eckhart										L.6		
Al(H2)	Valimet	2-5 μm							97.20%				

In general, the active aluminum content in the nanoaluminum powders varied from lot-to-lot. For the 80 nm Al, the active Al content ranged from 71 to 87%. This variation was due to production and passivation issues at the manufacturer. Another significant factor was the particle size distribution. In one lot of 80 nm Al, larger particles resulted in poor performing ENCs. This makes it critical for the manufacturer to run TGAs and hydrolyses on each lot as a quality check.

Appendix F: Ball Milling Process of Micron Oxidizers

Procedure:
1) A 200 ml Ceramic Mill Jar was charged with 50-60 grams of a micron metal oxide, 25 Alumina balls (0.5" ~2.56g/ball) and 150 ml of hexane.
2) The metal-oxide powder was milled on a U.S. Stoneware roller set at 20% power (~ 15 rpm) for 4 days.
3) The product was transferred into a 500 ml beaker, the milling balls removed and the product isolated by filtration through Whatman #5 filter paper.
4) The product was dried under vacuum for 1 h.

Appendix G: Ball Milling of Low-Density Ammonium Nitrate

Procedure:

1) A 200 ml Ceramic Mill Jar was charged with 50 grams of 1.7 mm LDAN, 25 Alumina balls (0.5" ~2.56g/ball), 0.5 grams of Palmitic Acid and 125 ml of hexane.
2) The metal-oxide powder was milled on a U.S. Stoneware roller set at 20% power (~ 15 rpm) for 4 days.
3) The product was transferred into a 500 ml beaker, the milling balls removed and the product isolated by filtration through Whatman #5 filter paper.
4) The product was dried under vacuum for 1 h.
5) The AN product was sieved through a series of 40, 60, and 150 mesh screens. The -60 and -150 AN products were outstanding for LFEP application.

Appendix H: Characterization of Oxidizer Powders

	Vendor		NAVAIR WD			
Sample	Source	Size	BET Size	BET Surface Area m^2/g	Decomposition Temperature (°C)	Appendix L SEM
MoO_3	Climax Molybdenum EM-NTO U2	nano	41 nm	28.6		L.10
MoO_3	Mallinckrodt Baker, Inc.		1.8 μm	0.699		L.11
Bi_2O_3	Aldrich	nano	322 nm	2.093	435-850	L.7
Bi_2O_3	Skylighter	Micron	2.5 μm	0.273		L.8
Bi_2O_3	Sigma-Aldrich	< 10 μm				L.9
CuO	Technanogy	nano	30 nm	32.2		
$AgIO_3$	Baker		400 μm			
$AgIO_3$	Baker Sieved		5-37 μm			
$AgIO_3$	Noah BM		1.75 μm			L.12
$AgIO_3$	City 09E22		1.2 μm			L.14
$AgIO_3$	Baker BM		895 nm	1.212		L.13
$AgIO_3$	City 01E56		861 nm			
$AgIO_3$	City 09E22 BM		632 nm			L.15
$AgIO_3$	CJ1831-96		474 nm			L.16
$AgIO_3$	CJ1831-69		320 nm			
$AgIO_3$	CJ1888-37		277 nm			
$AgIO_3$	CJ1788-94		275 nm			
$AgIO_3$	CJ1831-13B		271 nm			
$AgIO_3$	CJ1888-34		266 nm			
$AgIO_3$	CJ1888-33		236 nm			
$AgIO_3$	CJ1694-57		235 nm			
$AgIO_3$	CJ1831-13		181 nm			
$AgIO_3$	CJ1831-68		156 nm	6.92		L.17
I_2O_5	B&A		25 μm			
I_2O_5 BM	KTHW28		1.68 μm	7.41		

		800 nm	1.565		
Cu(IO$_3$)$_2$-H$_2$O					
Zn(IO$_3$)$_2$-H$_2$O			0.167		
Bi(IO$_3$)$_3$		62 nm		425-600	
Cu(IO$_3$)$_2$-H$_2$O BM	KTHW61	750 nm			
Bi(IO$_3$)$_3$	CJ1694-75	62 nm			
Low Density AN	Dyno Nobel	1.7 mm			
AN	W89 AN (BM)	900 nm	0.388		L.18

In Figure H.1, the TGA of the new oxidizer Bi(IO$_3$)$_3$ is shown. The thermal decomposition of the oxidizer appears to be heating rate dependent. At heating rates of 20 and 50° per minute, a weight loss was observed at 370°C and 500°C while for 1 and 5° per minute the major weight loss was seen at around 500°C. The Bi(IO3)3 decomposes into Bi$_2$O$_3$, O$_2$ and I$_2$.
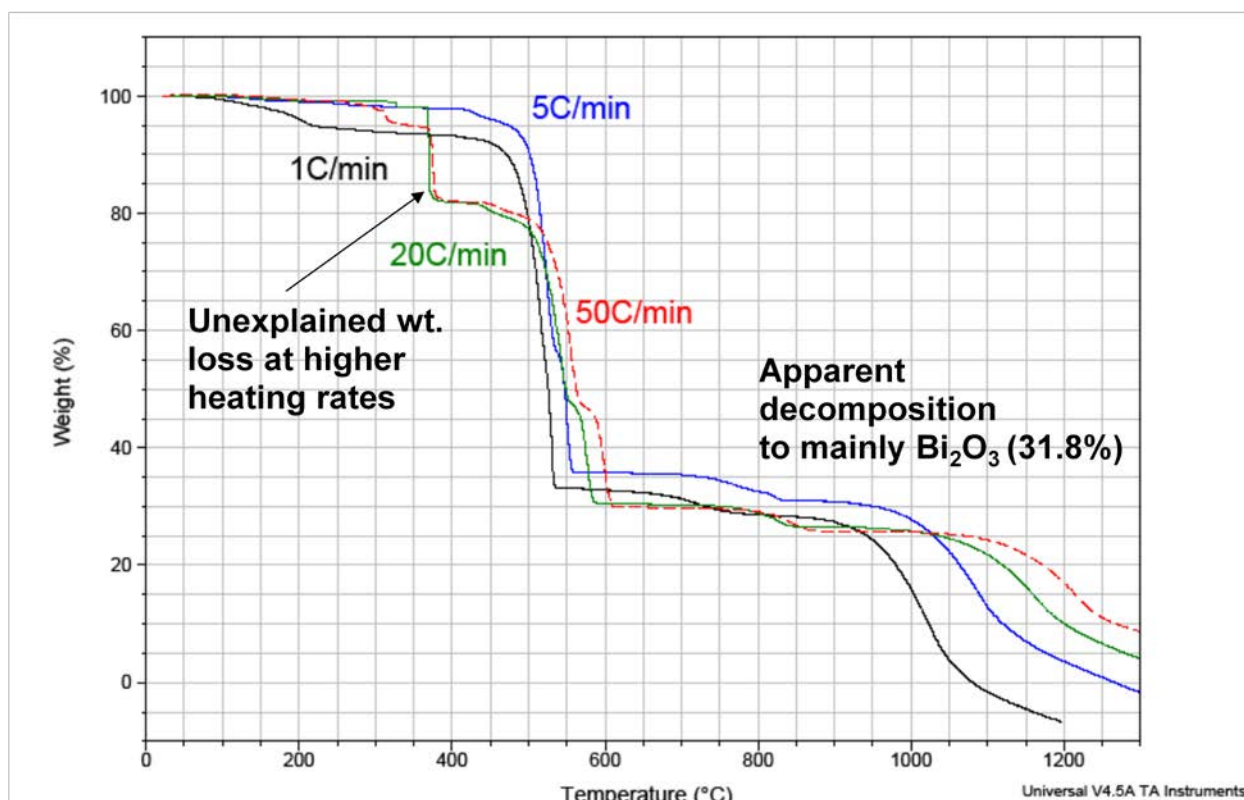
Figure H.1: TGA of Bi(IO$_3$)$_3$ Oxidizer

Appendix I: Electrostatic Discharge Sensitivities

	Energy (mJ)		
Aluminum Powders	No-Fire	Onset	50% Point
Al(33nm)	2.500	10.000	15.600
Al(50nm)	0.600	2.500	2.500
Al(80nm) Not Aged		0.090	0.090
Al(80nm) Aged in Air	10.000	15.600	22.500
ALEX(150nm)	10.000	15.600	22.500
Composites			
Al(80nm)M2210/MoO$_3$(45nm)		0.025	
Al(80nm)M2210/Bi$_2$O$_3$(50nm)	0.004	0.009	0.009
Aged Al(80nm)M2210/Bi$_2$O$_3$(50nm)	0.004	0.025	
Al(80nm)M2210/Bi$_2$O$_3$ (320nm)		0.025	
Aged Al(80nm)M2210/Bi$_2$O$_3$ (320nm)	0.100	0.225	0.225
Al(80nm)M2210/Bi$_2$O$_3$ (2.5 μm)	0.025	0.049	
Aged Al(80nm)M2210/Bi$_2$O$_3$ (2.5 μm)	0.400	0.900	
Al(80nm)M2210/Fe$_3$O$_4$ CJ1888-19A	0.100	0.400	0.900
Al(80nm)M2210/WO$_3$ (nano)		0.004	
Al(80nm)M2671/AgIO$_3$ AE90	0.004	0.009	0.025
Al(80nm)M2210/Teflon(1 μm)	40.000	44.100	44.100
Al(80nm)/ZnO (micron)	2500		
Al(80nm)/Cu(IO$_3$)$_2$·H$_2$O (750 nm)	1.600	2.500	
Al(80nm)/Zn(IO$_3$)$_2$·H$_2$O	4.225	4.900	
Al(80nm)/Bi(IO$_3$)$_3$·H$_2$O	0.100	0.144	
Al(H5)/Bi$_2$O$_3$(320nm)	2500		

The ESD data was collected on a ESD Tester built at China Lake to measure sensitivities in the micro joule range. The ESD sensitivities of nano-aluminum powders as received from the vendor under argon are very high. The 80 nm Al initially gave an onset and 50% fire point of 90 μJ but after aging in air for 6 weeks, the onset and 50% fire points jump up to 15.6 and 22.5 mJ, respectively. The thickening of the oxide coating reduces the Al ESD sensitivity by almost a factor of 200. However, the ESD sensitivity of the Al(80nm)/Bi$_2$O$_3$(50nm) composite increased by only a factor of 3 after aging the nano Al powder. The ESD sensitivities of ENCs are dependent on the oxidizer, oxidizer particle size, and Al powder. Every composite containing nano aluminum showed some ESD sensitivity except Al(80nm)/ZnO. The reason is not known but may be due to the higher electrical conductivity of micron sized ZnO.

Appendix J: Bulk ENC Preparation by Modified Ball Milling

The ball milling of mixtures of micron aluminum and oxidizer powders are well documented in the literature. Under standard ball milling conditions, even micron composites begin to react after a certain level of milling and milling time (< 4 h). Due to the high surface area and high impact and friction sensitivity of nano energetic composites, conventional ball milling may be extremely hazardous. To mitigate some of the hazard, mixtures of nano fuels and nano oxidizers were ball milled in HD polyethylene container. Scale sizes started at 2 gram and were increased to over 100 grams per batch. The milling time, ball to product ratio (BPR), milling media (alumina and steel), and milling media size were investigated. The Al Pan Dent Test was used to monitor the material during milling. A US Stoneware roller was used for all milling experiments. A summary of Ball Milling Experiments is found in Table J.1

Table J.1: Ball Milling Runs

Sample Number	Aluminum	Oxidizer	Wt% Al	BPR	Al/Oxid	Milling Time (h)	Deflection (mm/g)	Scale	Milling Balls
Z2	Al(50nm)	MoO_3 (45nm)	49.0%	2.14	3.28	24	113	12	C 0.5"
X67	Al(80nm) M2453	MoO_3 (45nm)	33.7%	2.56	2.20	23	200	10	C 0.5"
Z5	Al(80nm) M2210	MoO_3 (45nm)	39.0%	2.55	2.52	17	154	10	C 0.5"
AF139	Al(80nm) M2210	MoO_3 (45nm)	40.5%	4.00	2.69	72	212	100	SS 5 mm
Z18	Al(80nm) M2453	MoO_3 (45nm)	39.0%	1.28	2.76	17	132	20	C 0.5"
AF20	Al(80nm) M2671	MoO_3 (45nm)	40.0%	10.00	2.76	24	187	10	SS 5 mm
AF14	Al(80nm) M2671	MoO_3 (45nm)	40.0%	0.26	2.8	96	219	100	C 0.5"
AF16	Al(80nm) M2671	MoO_3 (45nm)	40.3%	4.10	2.8	36	216	100	SS 5 mm
AF17	Al(80nm) M2671	MoO_3 (45nm)	40.4%	4.07	2.8	72	212	101	SS 5 mm
AD27	Al(80nm) M2210 New	MoO_3 (45nm)	40.5%	0.26	2.87	84	173	100	C 0.5"
AB2	Al(80nm) M2453	MoO_3 (45nm)	40.3%	1.05	2.92	24	167	24	C 0.5"
Z43	Al(80nm) M2210	Bi_2O_3 (50nm)	15.0%	2.56	2.25	8	417	10	C 0.5"
AE77	Al(80nm) M2671	$AgIO_3$ (236nm)	22.0%	4.00	2.51	14	970	100	SS 5 mm

AE84	Al(80nm) M2671	AgIO$_3$ (266nm)	21.8%	4.00	2.48	16	1100	100	SS 5 mm
AE85	Al(80nm) M2671	AgIO$_3$ (266nm)	21.8%	4.00	2.51	24	1091	100	SS 5 mm
AE86	Al(80nm) M2671	AgIO$_3$ (266nm)	21.8%	4.00	2.51	16	1112	100	SS 5 mm
AE95	Al(80nm) M2671	AgIO$_3$ (277, 276, 271nm)	20.0%	4.00	2.54	24	1291	100	SS 5 mm
AE87	Al(80nm) M2671	AgIO$_3$ (277nm)	21.8%	4.00	2.51	7	1235	100	SS 5 mm
AE88	Al(80nm) M2671	AgIO$_3$ (277nm)	21.8%	4.00	2.51	22	1230	100	SS 5 mm
AE89	Al(80nm) M2671	AgIO$_3$ (277nm)	21.8%	4.00	2.51	24	1308	100	SS 5 mm
AE90	Al(80nm) M2671	AgIO$_3$ (277nm)	21.8%	4.00	2.51	23	1297	100	SS 5 mm
AE91	Al(80nm) M2671	AgIO$_3$ (277nm)	21.8%	4.00	2.51	24	1257	100	SS 5 mm
AE92	Al(80nm) M2671	AgIO$_3$ (277nm)	22.2%	4.00	2.54	24	1291	100	SS 5 mm
AE73	Al(80nm) M2671	AgIO$_3$ (474nm)	22.0%	4.00	2.50	6	1236	100	SS 5 mm
AE53	Al(80nm) M2210	AgIO$_3$ (632nm)	23.0%	4.10	2.32	24	1035	100	SS 5 mm
AE72	Al(80nm) M2671	AgIO$_3$ (632nm)	23.0%	4.10	2.65	5	1031	100	SS 5 mm
AB68	Al(Alex)	MoO$_3$ (45nm)	33.0%	2.56	2.23	96	175	10	C 0.5"
AB63	Al(Alex)	MoO$_3$ (45nm)	35.0%	2.56	2.44	72	183	10	C 0.5"
AB72	Al(Alex)	MoO$_3$ (45nm)	36.0%	2.56	2.55	120	185	10	C 0.5"
AB60	Al(Alex)	MoO$_3$ (45nm)	37.0%	2.56	2.67	75	223	10	C 0.5"
AB70	Al(Alex)	MoO$_3$ (45nm)	38.0%	2.40	2.77	120	208	10.65	C 0.5"
AB58	Al(Alex)	MoO$_3$ (45nm)	39.0%	2.56	2.89	39	166	10	C 0.5"
Z82	Al(Alex)	MoO$_3$ (45nm)	40.0%	2.56	3.02	72	158	10	C 0.5"
AB77	Al(Alex)	AgIO$_3$ (156nm)	20.0%	5.12	2.23	63	756	10	C 0.5"
AB78	Al(Alex)	AgIO$_3$ (320nm)	20.0%	5.12	2.23	75	769	10	C 0.5"

The Ball to Product Ratio (BPR) was varied from 0.25 to over 5 and only affected the amount of milling time required. The ball milling material did not impact the material performance. Both alumina and stainless steel milling balls gave high performing NECs. However, the alumina balls were found to pick up charge during the milling process and resulted in 2 separate fires during the isolation of the products. The use of ceramic milling balls capable of picking up charge by triboelectric charging is not advisable. For the Al(80nm)/AgIO$_3$ NEC system, there appears to be an optimum AgIO$_3$ particle size centered around 277 nm. For the Alex/MoO$_3$(45nm) NEC system, the optimal Al/MoO$_3$ molar ratio was 2.67 and gave a Deflection of 223 mm/g. The ball milled NEC products are in many cases superior to small scale sonication materials with the same composition.

Al(50nm)/MoO$_3$(45nm) – The 250 ml PE milling bottle was charged with 5.86 g of 50 nm Al, 6.10 g of MoO$_3$, 53 ml of hexane and 10 alumina milling balls (0.5" diameter, 25.6 g). The composite composition was 49 wt% Al(50nm) and 51 wt% MoO$_3$ that corresponds to a 3.28 Al/MoO$_3$ molar ratio. The BPR was 2.14. The roller was set to 50% power and aliquots were removed periodically and evaluated using the Al Pan Dent Test. The Deflection versus milling time is shown in Figure J.1.
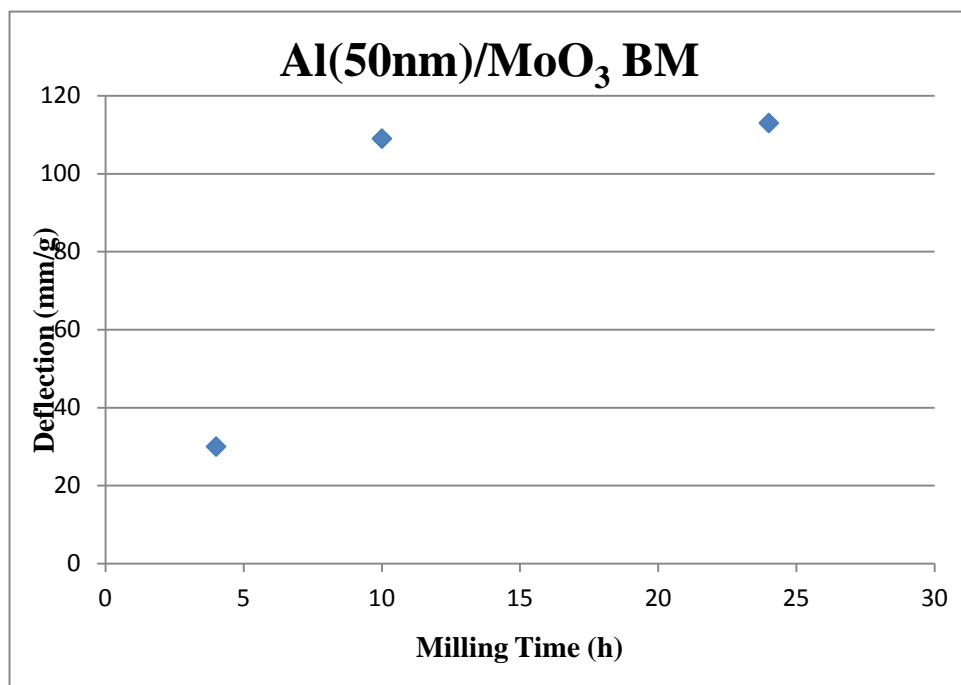
Figure J.1: Deflection Versus Milling Time for Ball Milling Preparation of Al(50nm)/MoO$_3$

After 24 h the milling was stopped and a "Deflection" of 113 mm/g was obtained which was consistent with KTHY7H (112 mm/g) that was prepared by sonication.

In Figure J.2, the Deflection versus milling time is graphed. The sample was ball milled for only 8 hours using ceramic balls. It was later determined that from other ball milling experiments that a minimum milling time of 72 hours is needed to optain maximum deflections.
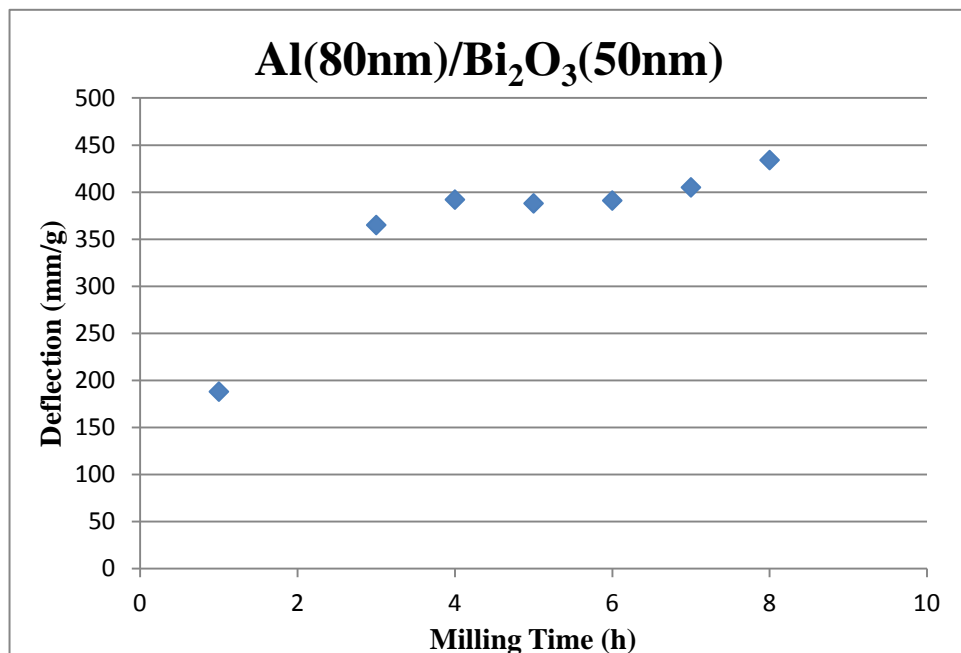
Al(80nm)/Bi$_2$O$_3$(50nm)

Figure J.2: Deflection Versus Mill Time for Al(80nm)/Bi$_2$O$_3$(50nm)

Al(80nm)M2453/MoO$_3$
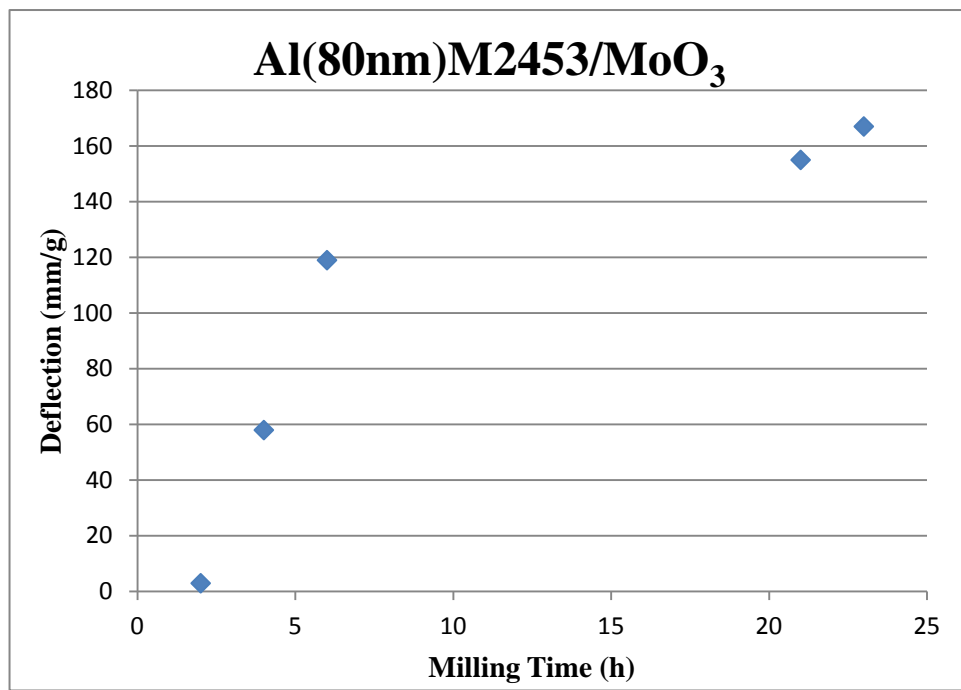
Figure J.3: Deflection Versus Mill Time for Al(80nm)/MoO$_3$(45nm)

In Figure J.4, the Deflection versus milling time is shown for the AlEX/AgIO$_3$ composite. This composite exhibits only about 70-75% of the deflection observed for the Al(80nm)/AgIO$_3$ ball milled samples.
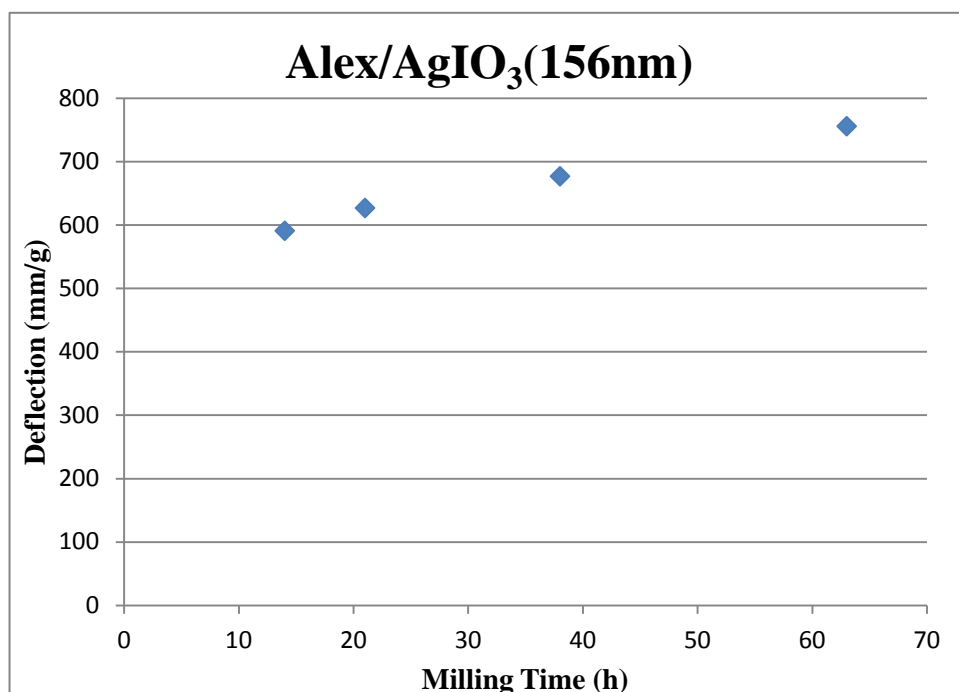
Alex/AgIO$_3$(156nm)

Figure J.4: Deflection Versus Mill Time for Al(80nm)/AgIO$_3$(156nm)

The 150nm aluminum powder (ALEX) from Argonide was investigated as a lower cost alternative to 80 nm Al. The Alex/MoO$_3$ NEC was optimized using ball milling samples rather than by sonication because the sonicated samples performed poorly. The optimization of Alex/MoO$_3$(45nm) by sonication and modified ball milling is shown in Figure J.5:

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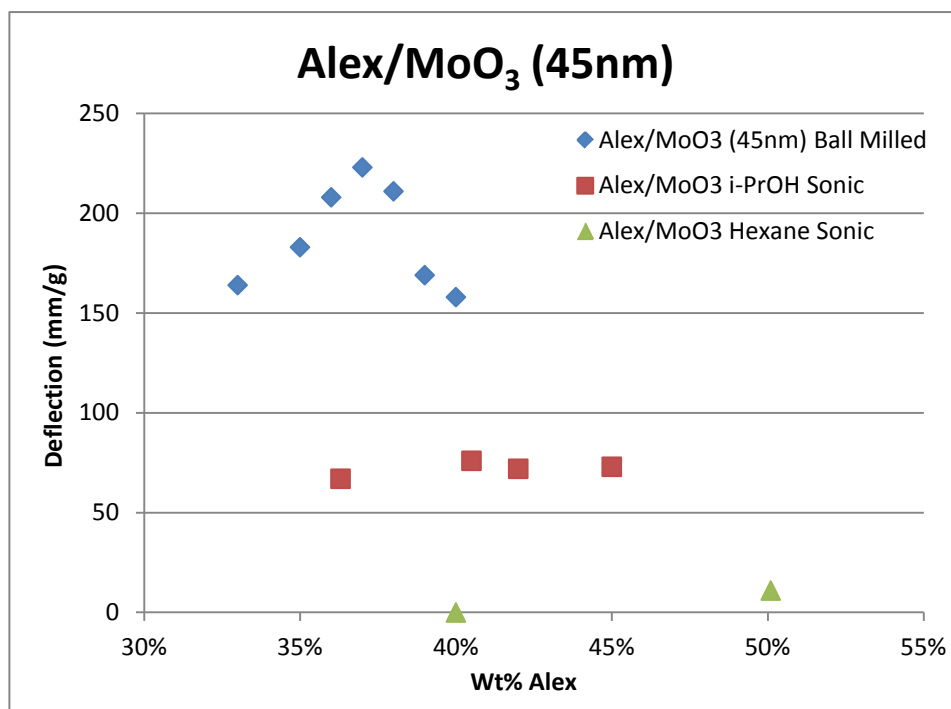
Figure J.5: Alex/MoO$_3$(45nm) NECs Made By Sonication and Ball Mill

Based on the hydrolysis data, Alex is 85% active Al and the optimized formulation (AB60) with a 37/63 Alex to MoO$_3$ weight ratio corresponds to a 2.68 to 1 molar ratio. The Al(80nm)/MoO$_3$ NEC optimized to a 2.67 molar ratio by sonication which may or may not be coincidence. The Deflection for the Alex/MoO$_3$ NEC (AB60) is slightly better than the best Al(80nm)/MoO$_3$ NEC (AF14). Prior to completing the optimization of Alex/MoO$_3$, a LFEP using the 39/61 Al(Alex)/MoO$_3$ NEC (AB58) was made. The Alex based LFEP contained 64.6% AB58, 31.4% AN, 2% Kel-F and 2% C and gave an average ambient AUR-AT of 3.67 ± 0.43 ms and a LT AUR-AT of 4.88 ± 2.26 ms. The high ambient AUR-ATs were probably due to the fact that the Alex/MoO$_3$ NEC was too fuel rich. In all probability, the optimized 37/63 Alex/MoO$_3$ (AB60) based LFEP would have produced much better ambient AUR-ATs. However, the LT AUR-ATs was potentially an issue since Alex contains only small quantities of sub 80 nm particles. The low percentage of small particles may lead to an ignition delay. The Alex based LFEP was discontinued by direction of the ESTCP review board.

APPENDIX K: Deflections for Energetic Nanocomposites

Name	Composite	Al/Oxidizer Ratio	Wt Ratio	Deflection (mm/g)	Method
Y7H	Al(50nm)/MoO$_3$(45nm)	3.25	49/51	112	Sonic
Z2	Al(50nm)/MoO$_3$(45nm)	3.25	49/51	113	BM
W46F	Al(80nm)/MoO$_3$(45nm)	2.67	38.6/61.4	156	Sonic
AF14	Al(80nm)/MoO$_3$(45nm)	2.63	40/60	217	BM
AF53	Al(80nm)/MoO$_3$ (1.6 μm)	2.63	40/60	0	Sonic
AF111	Al(80nm)/Bi$_2$O$_3$ (50 nm)	2.30	15.2/84.8	548	Sonic
Y17	Al(80nm)/Bi$_2$O$_3$(320nm)	2.27		513	Sonic
AB3	Al(80nm)/Bi$_2$O$_3$(320nm)	2.26		486	BM
Y17	Al(80nm)/Bi$_2$O$_3$ (320 nm)	2.27	15.1/84.9	513	Sonic
U40	Al(80nm)/Bi$_2$O$_3$ (1.5 μm) Skylighter	2.19	14.6/85.4	408	Sonic
Z84B	Al(80nm)/Bi$_2$O$_3$ (2.5 μm) Skylighter	2.26	15.0/85.0	140	Sonic
AF109	Al(80nm)/Bi$_2$O$_3$ (10 μm) Sigma	2.46	16.1/83.9	166	Sonic
AF125	Al(80nm)/Bi$_2$O$_3$ (Atlantic Equip. Eng < 10 μm)	2.17	14.5/85.5	113	Sonic
Z67	Al(80nm)/AgIO$_3$(181 nm)	2.12		1124	Sonic
AD32	Al(80nm)/AgIO$_3$(271 nm)	2.14		1095	Sonic
AE80	Al(80nm)/AgIO$_3$(277 nm)	2.19		1308	BM
AF15	Al(80nm)/AgIO$_3$ (680 nm) City Chemical (BM)	2.31	23.0/77.0	1007	Sonic
AF23	Al(80nm)/AgIO$_3$ (895 nm) W55 Baker	2.26	22.5/77.5	468	Sonic
AD35	Al(80nm)/AgIO$_3$ (1.6 μm) City Chemical	2.11	21.4/78.6	784	Sonic
AE27	Al(80nm)/AgIO$_3$ (1750 nm) Noah BM -400	2.26	22.4/77.6	467	Sonic
AE137	Al(80nm)/NiO (Baker)	0.67	25.5/74.5	0	Sonic
Z36	Al(80nm)/NH$_4$IO$_3$(micron)	2.25		14	Sonic
AB5A	Al(80nm)/CuO(40nm)	0.72		228	Sonic
AB14	Al(80nm)/Ag$_2$MoO$_4$	2.75		221	Sonic

AA64	Al(80nm)/Ag$_2$O (< 1 μm)	0.71	21.1/78.9	0	Sonic
BH1858-93G	Al(80nm)/Bi(IO$_3$)$_3$	9.05	31.6/68.4	736	Sonic
AB15	Al(80nm)/I$_2$O$_5$	4.82	34.5/65.5	36	Sonic
AF114	Al(80nm)/I$_2$O$_6$	4.1	30/70	276	Sonic
AD51	Al(80nm)/Fe$_2$O$_3$ MACH1	2.52	36.5/63.5	0	Sonic
AB68	ALEX/MoO$_3$(45nm)	2.24	33/67	175	BM
AB63	ALEX/MoO$_3$(45nm)	2.45	35/65	183	BM
AB72	ALEX/MoO$_3$(45nm)	2.56	36/64	185	BM
AB60	ALEX/MoO$_3$(45nm)	2.68	37/63	223	BM
AB70	ALEX/MoO$_3$(45nm)	2.78	38/62	211	BM
AB58	ALEX/MoO$_3$(45nm)	2.91	39/41	166	BM
Z75	ALEX/MoO$_3$(45nm)	3.02	40/60	11	Sonic
Z82	ALEX/MoO$_3$(45nm)	3.02	40/60	158	BM
AD23	ALEX(150nm)/MoO$_3$ (45 nm)	3.1	40.5/59.5	76	Sonic
Z79	ALEX/MoO$_3$(45nm)	4.54	50/50	11	Sonic
Z87	ALEX/Bi$_2$O$_3$(320nm)	2.35	13.8/86.2	469	Sonic
Z81	ALEX/Bi$_2$O$_3$(320nm)	2.60	15/85	526	Sonic
Z80	ALEX/Bi$_2$O$_3$(320nm)	2.76	15.9/84.1	397	Sonic
AB91	ALEX/Bi$_2$O$_3$(320nm)	2.60	15/85	483	BM
AD87	ALEX/AgIO$_3$ City	2.21	20/80	2	Sonic
AD88	ALEX/AgIO$_3$ City	2.78	24/76	156	Sonic
AD89	ALEX/AgIO$_3$ City	2.52	22/78	753	BM
AD96B	Al(138nm)/AgIO$_3$ (1.6 μm) City Chemical	24.6/75.4	2.9	1.45	507
AB89	nTi/Bi$_2$O$_3$ (320 nm)	14.3/85.6	1.62	1.08	58
AA93	nTi/AgIO$_3$ (271 nm)	20.7/79.3	1.54	1.03	454
	Pb (N$_3$)$_2$				1089
	Cu(N$_3$)$_2$				1088
	Lead Styphnate				0
	Mix FA-874 Used in the M52A3B1				0

	Primer					

The Al Pan Dent Test was used to characterize a number of known and new composites. Until the development of the Al Pan Dent Test, it was far too expensive to evaluate many thermite composites. The best energetic nanocomposite known previously was the Al/Bi$_2$O$_3$ that is the basis for the Army's small caliber lead-free primers and for the Navy's Lead-Free Cartridge Actuated Devices. Several conclusions have been made based on the Al Pan Dent Test data.

Al/Bi$_2$O$_3$ composites - The critical size of the Bi$_2$O$_3$ particle was determined to be 1.5 micron or less for the Al(80nm)/Bi$_2$O$_3$ composites. The deflection of the Al(80nm)/Bi$_2$O$_3$ ENC drops from 408 down to 140 mm/g by switching 1.5 micron Bi$_2$O$_3$ to 2.5 micron. The larger ALEX (150nm) Al appears to be a viable replacement for 80 nm Al in the Army's and Navy's lead-free munitions programs. ALEX is not only cheaper but also less ESD sensitive than 80 nm Al. Much safer Al/Bi$_2$O$_3$ composites could be made.

Highest Performance - By far, the best composite identified to date with a deflection number of 1308 mm/g is Al(80nm)/AgIO$_3$(277nm) made by ball milling.

Potential Improvement of the Standard LFEP - Although the Al/MoO$_3$ ENC was one of the best ENCs at the start of the program, a number of superior composites have been identified. A simple one-to-one replacement of the Al/MoO$_3$ in the **Standard LFEP** formulation with a superior ENC should improve the performance.   The Al(80nm)/Bi(IO$_3$)$_3$ composite is currently the top ENC replacement for Al/MoO$_3$ in the LFEP.

Ball Milling - Some composites made by the conventional ultra-sonication process produced poor performing composites but good composites when ball milled. The ball milling process may be improving the mixing by breaking up clumps of material more effectively than sonication. Almost every ENC showed superior performance when made by ball milling as compared to sonication, provided adequate milling time was used. The Al/Bi$_2$O$_3$ composites were the least explored by ball milling due to an accidental ignition. Intermediate deflection data was gathered and reported above.

Anomaly - Most ENCs using ALEX in place of 80 nm Al showed much poorer performance when made by the sonication process with the exception of the ALEX/Bi$_2$O$_3$(320nm) ENC. The cause is not understood.

Al Pan Dent Testing of Primary Materials - Several primary material were tested including lead azide, copper azide, lead styphnate and Mix FA-874 (used in the M52A3B1 primer). Lead styphnate and the Mix FA-874 both burned in a flash. The azide compounds both gave a sharp audio report and produced high deflection numbers over 1000 mm/g. The Al Pan Dent Test is not a suitable method for testing some materials. Confinement may be needed for some materials to produce a detonation or rapid deflagration.

The Technanogy 52 nm Al powder is shown in Figure L.1. It is highly agglomerated into micron sized clumps.
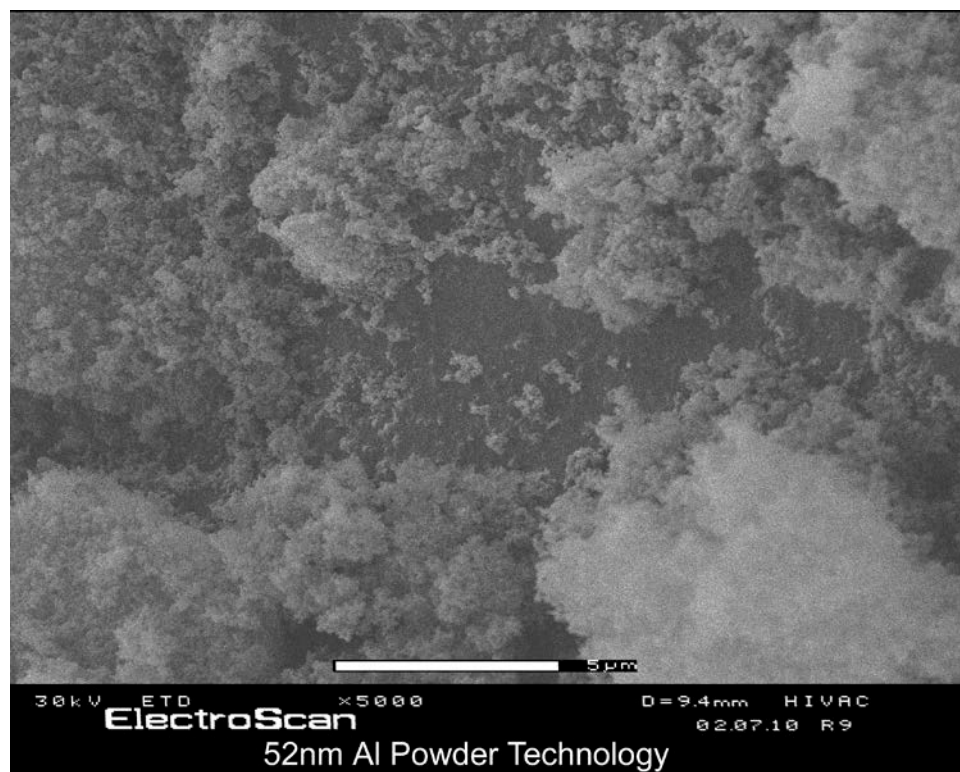
Figure L.1: Technanogy 52 nm Al Powder

Figure L.2 shown the SEM of the LANL 50 nm Al powder. In comparison to the Technanogy powder, it appears less agglomerated. The necking between particles suggests that these particles are more hard agglomerates (fused).
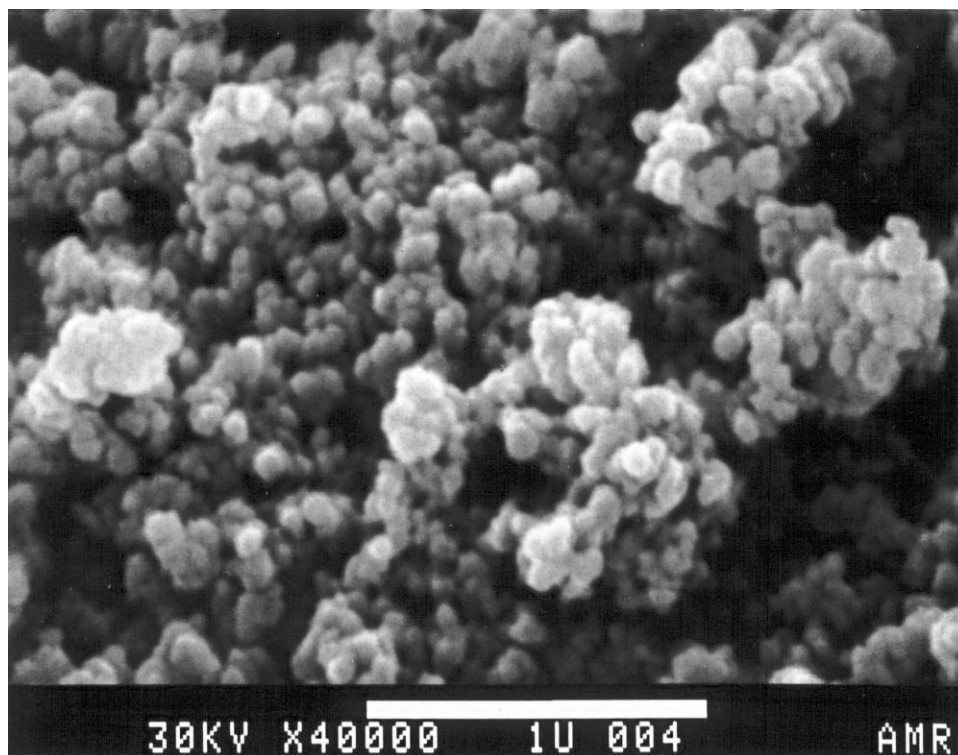
Figure L.2: Los Alamos 50 nm Al Powder LANL-A

Figure L.3 of Argonide's 150 nm Al (ALEX) appears to be similar to the LANL Al powder but with a larger particle size.
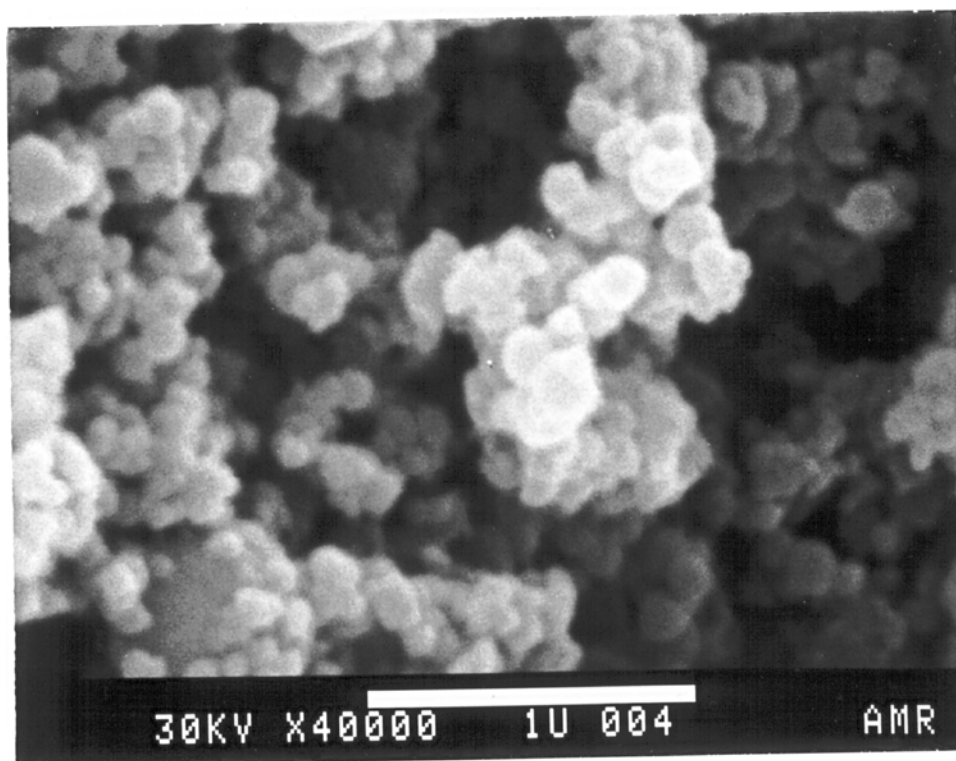
Figure L.3: Argonide's 150 nm Al Powder

Figure L.4 shows NovaCentrix's 80 nm Al. The particles appear to be more discrete but there are some larger sized particles.
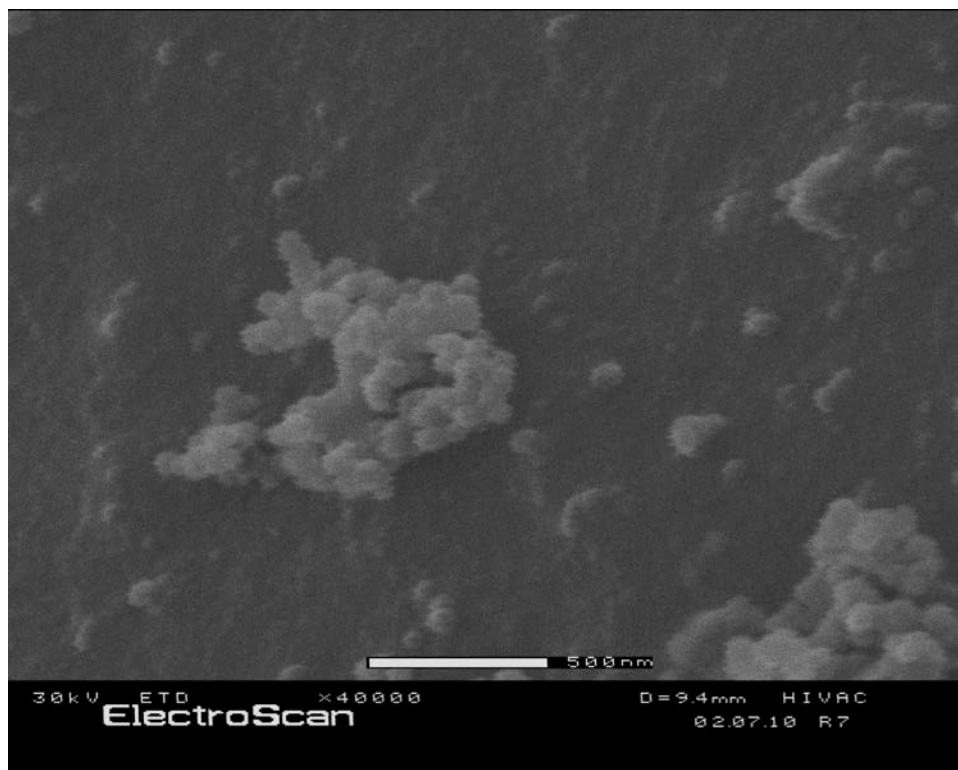
Figure L.4: NovaCentrix's 80 nm Al Powder

Figure L.5 is the SEM of Aldrich's nano titanium powder. The particles appear to be around 100 nm in size but there seems to be extensive hard necking between particles. The hard necking will reduce fuel-oxidizer mixing efficiency.
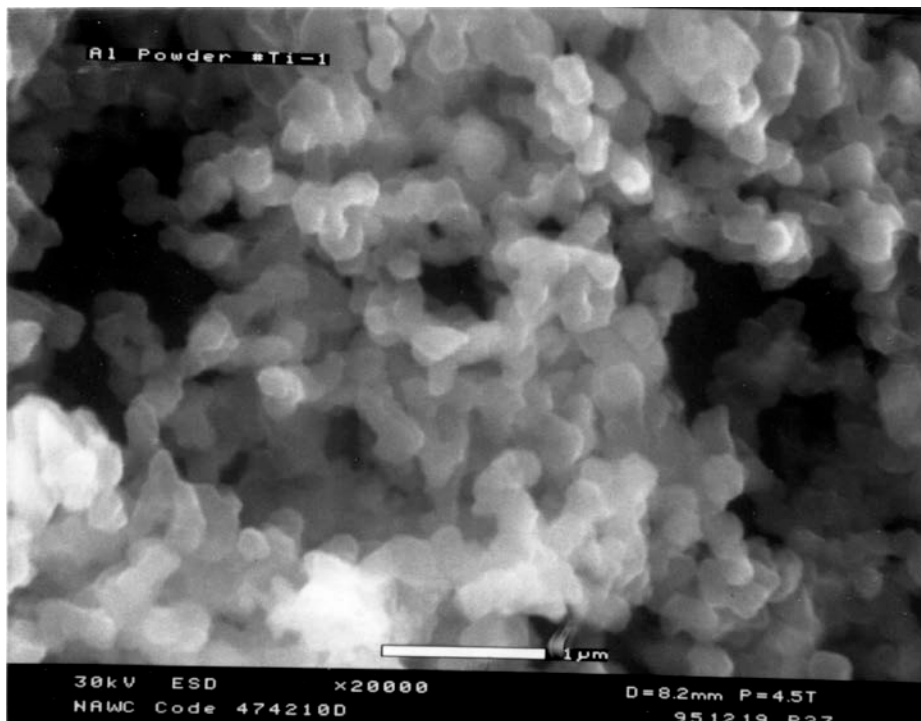
Figure L.5: Aldrich nano Ti Powder

In Figure L.6, Eckart's Flake Al appears to be a mixture of flakes and smaller oddly shaped particles. All attempts to make high performance ENCs using the Eckart Flake Al failed. The flake morphology may be reducing mixing efficiency with the oxidizer.
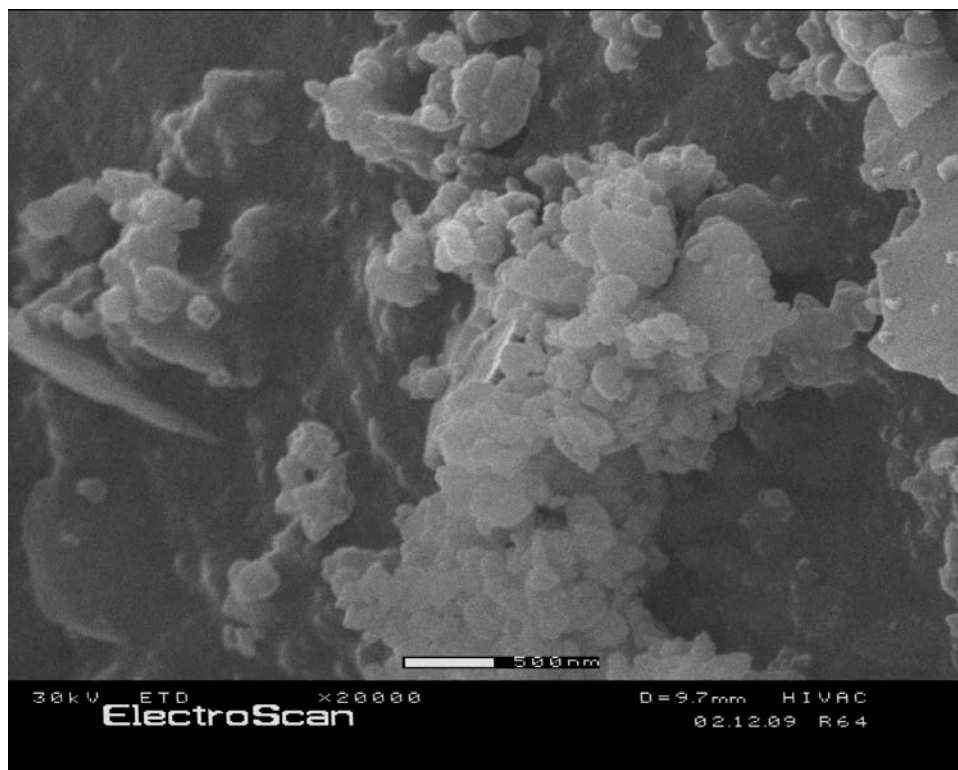
Figure L.6: Eckhart's Flake Al

The SEM of the Aldrich 320 nm Bi_2O_3 powder is shown in Figure L.7. The Bi_2O_3 particles are in general spherical but with a wide particle size distribution ranging from 50 nm to 1 micron.
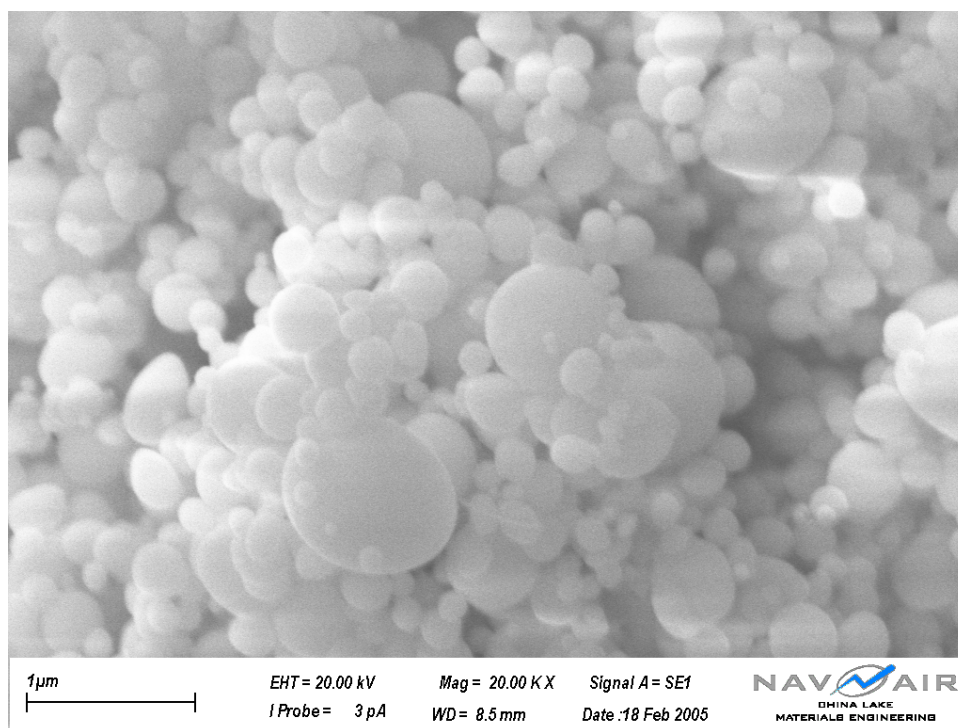
Figure L.7: Aldrich's 320 nm Bi_2O_3

Skylighter's Bi_2O_3 is shown in Figure L.8.   The particles appear logged shaped and has a wide range of particle sizes. Sharp flat surfaces are not observable suggesting poor crystallinity.
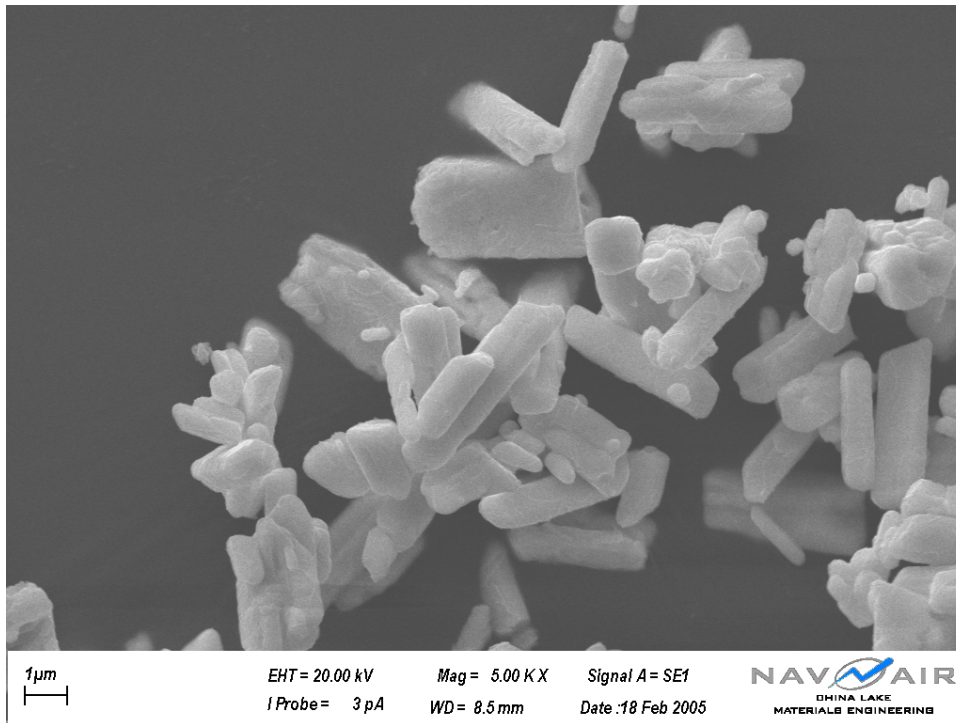
Figure L.8: Skylighter's Bi_2O_3

The Sigma-Aldrich 10 micron Bi_2O_3 is shown in Figure L.9. This material appears to be distinctly crystalline with many facets. Many of the crystals also appear to be fractured and twinned.
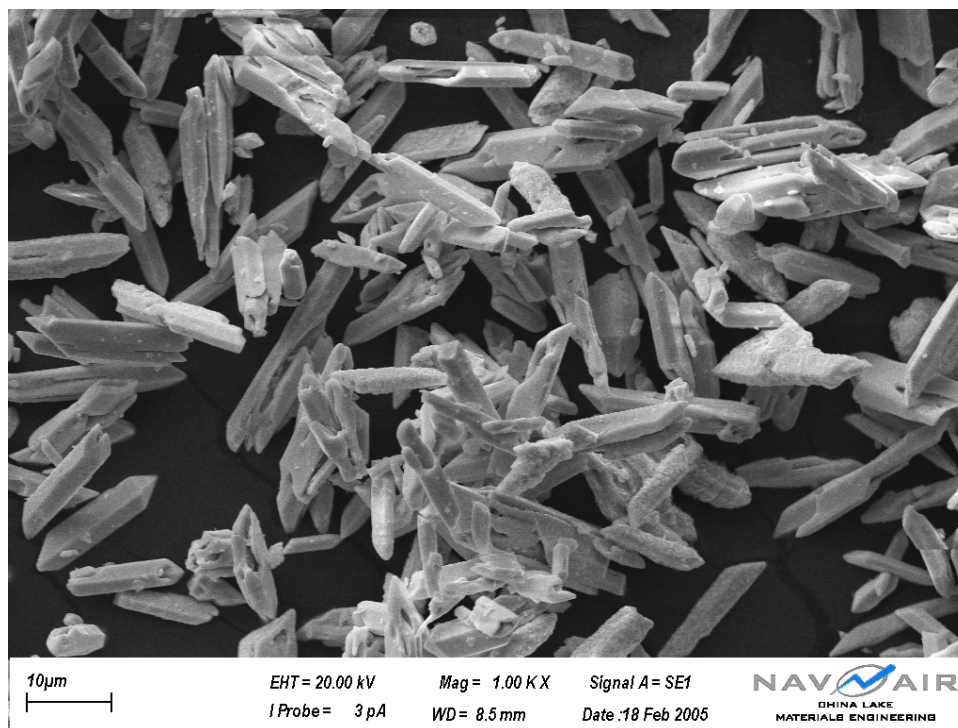
Figure L.9: Sigma-Aldrich's < 10 µm Bi_2O_3

The morphology of almost every Bi_2O_3 powder is different ranging from spherical to hexagonal crystals. At this point, the importance of the Bi_2O_3 morphology is not known. However, it appears that the average particle size is more important. The two batches of Skylighter Bi_2O_3 were 1.5 and 2.5 microns in size and Al(80nm) composites exhibited very different properties, the 1.5 micron gave good ENCs and the 2.5 gave poor ENCs.

The MoO_3 from Climax Molybdenum shown in Figure L.10 has at least three distinct morphologies, large flakes, small spheres and an amorphous phase. Some of the flakes are over a micron in length and are probably hexagonal MoO_3. The small spherical particles are believed to be monoclinic MoO_3. The amorphous material is probably $MoO_3 \cdot (H_2O)_{0.33}$ which disappears when the sample is heated to 200°C. The monoclinic phase can be converted into the orthorhombic phase when heated to 400°C for 4 hours but this leads to a sharp increase in particle size.
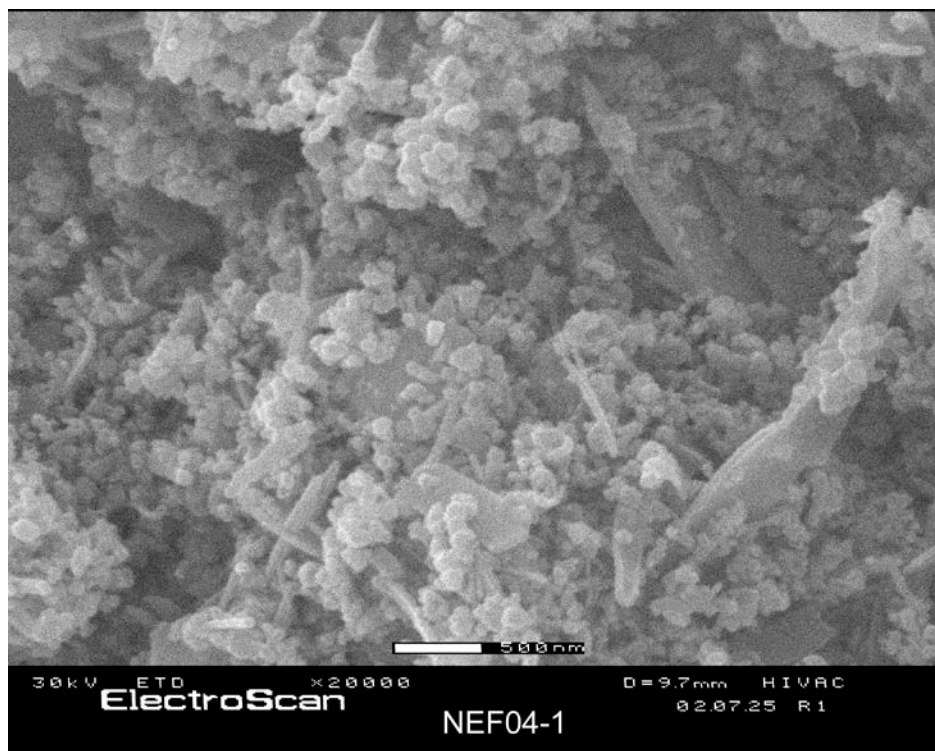
Figure L.10: Climax Molybdenum's MoO$_3$

Figure L.11 is the SEM of a Mallinkrodt sample that was ball milled to an average particle size of 1.5 microns. The sample is orthorhombic MoO$_3$ and the Al Pan Den Test deflections of the Al/MoO$_3$ composites are poor. The particle size is believed to be too large.
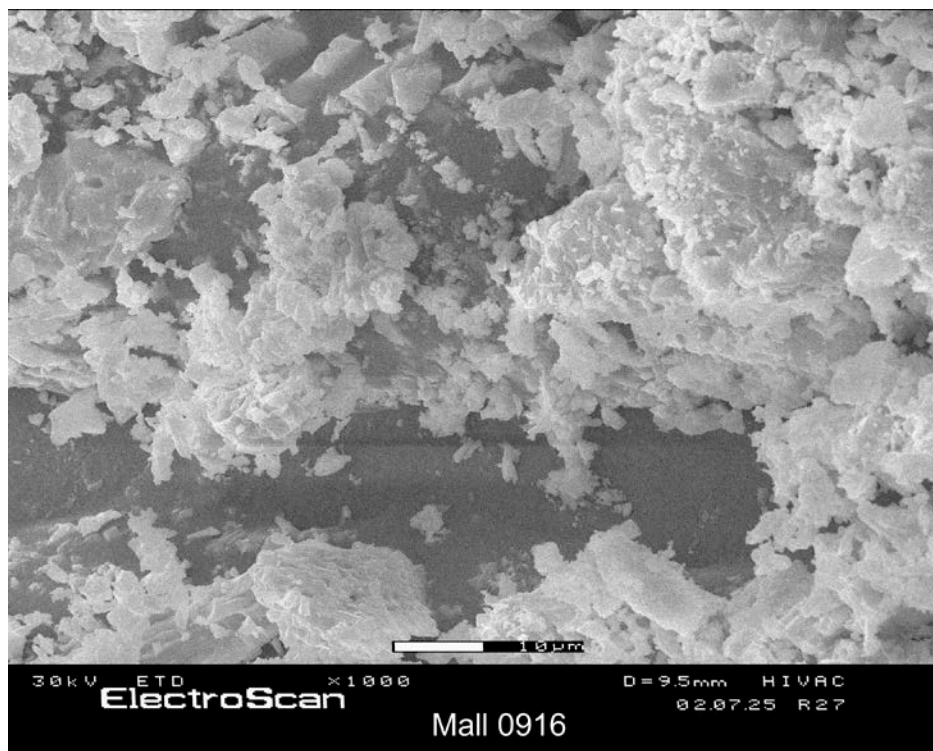
Figure L.11: Mallinkrodt's MoO_3

Figures L12 to L.16 are SEMs of $AgIO_3$ from Noah Tech, B&A, City Chemical and $AgIO_3$ prepared in-house. The Noah Tech sample in Figure L.12 is 1.75 microns in size and appears to be amorphous.
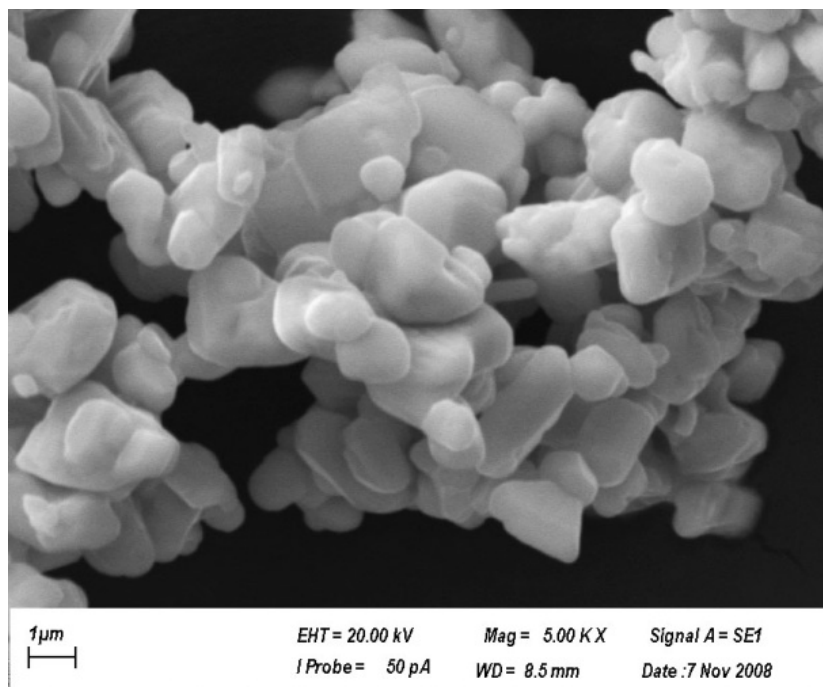
Figure L.12: Noah Tech 1.75 μm $AgIO_3$

The B&A AgIO$_3$ in Figure L.13 has been ball milled but it appears to have a wide particle size distribution ranging from 0.2 to 10 microns.
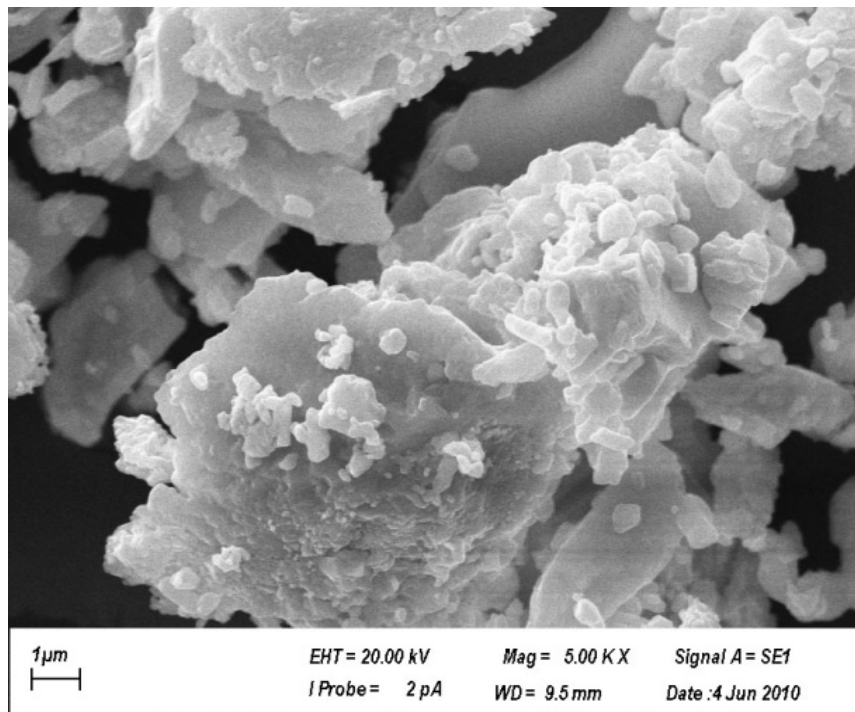
Figure L.13: B&A's AgIO$_3$ After Ball Milling

The City Chemical AgIO$_3$ shown in Figure L.14 has an average particle size of 1.6 microns. The Al(80nm)/AgIO$_3$ composites using the City Chemical sample gave good deflection data (up to 768 mm/g).   The morphology is similar to Noah Tech's but better performing.
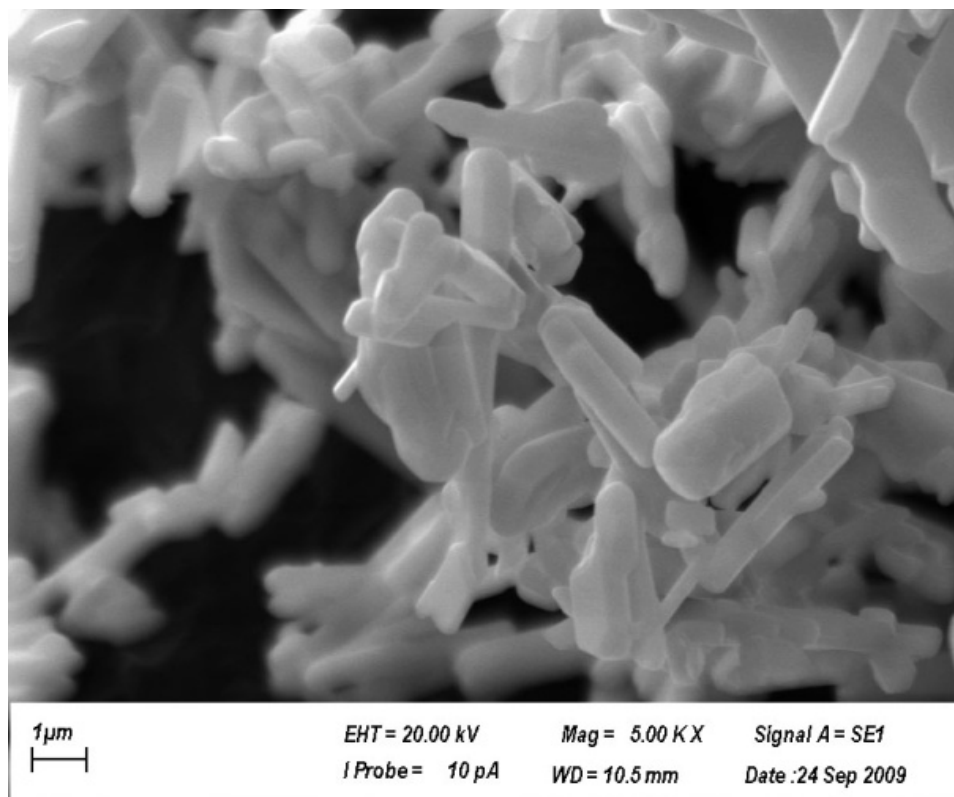
Figure L.14: City Chemical's 1.6 μm AgIO$_3$

Figure L.15 shows the City Chemical AgIO$_3$ after ball milling. The average particle size dropped from 1.6 microns down to 632 nm. The sample still shows a wide distribution of particle sizes but the deflection of the Al(80nm)/AgIO$_3$ composite improved to 1007 mm/g. This is currently the highest deflection observed for a commercially available oxidizer source.
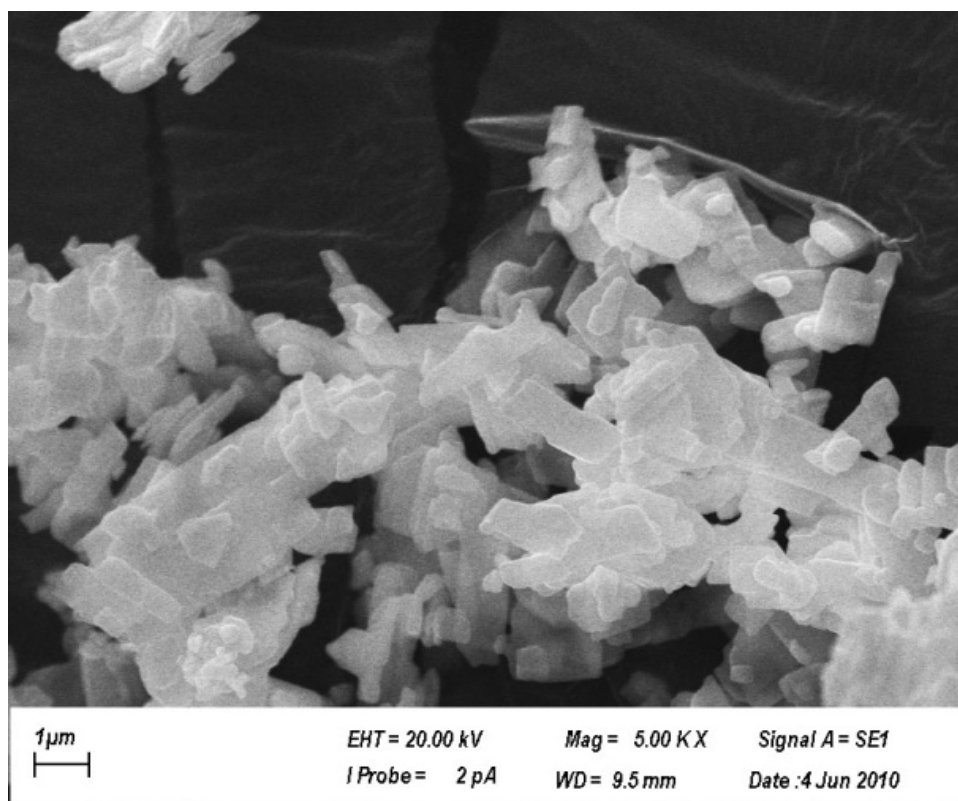
Figure L.15: Ball Milled City Chemical 632 nm AgIO$_3$

Figure L.16 is the SEM of AgIO$_3$ made from the solution reaction between AgNO$_3$ and NaIO$_3$ in water. The average particle size was determined to be 474 nm by BET. The particles appear to be fused plates.
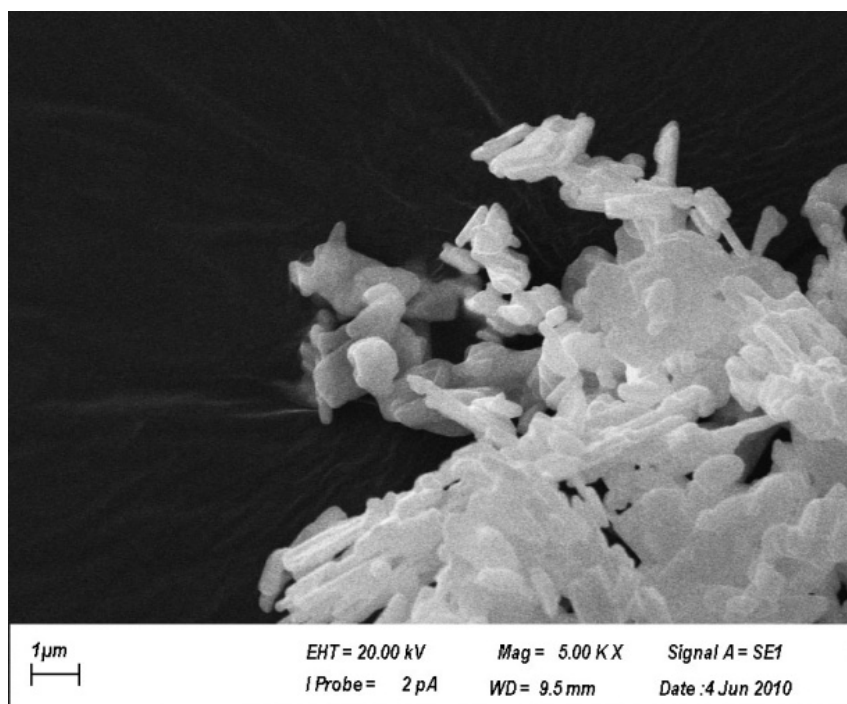
Figure L.16: NAVAIR's 474 nm $AgIO_3$

Figure L.17 is the SEM of $AgIO_3$ made by China Lake's solution process. A more dilute solution was used to prepare $AgIO_3$ with an average particle size of 156 nm. In reality, the products are thin plates and the BET calculation converts the surface area into spheres with equivalent surface areas.
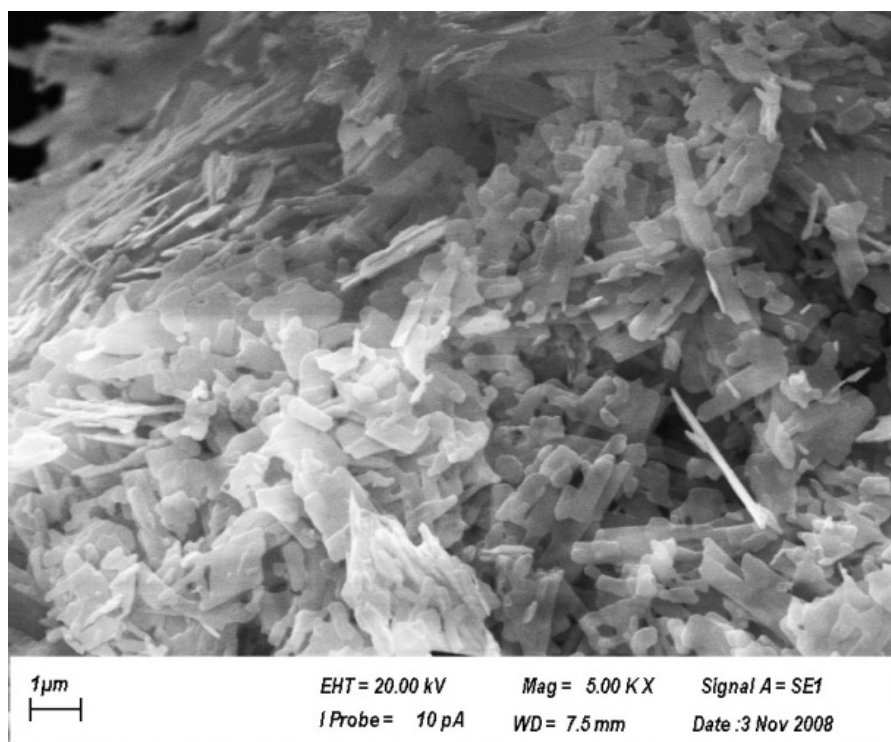
Figure L.17: NAVAIR's 156 nm $AgIO_3$

Figure L.18 is the SEM of Ag_2O prepared from the reaction of $AgNO_3$ with KOH followed by dehydration. The SEM shows a wide particle size distribution ranging from 50 nm to 5 microns.
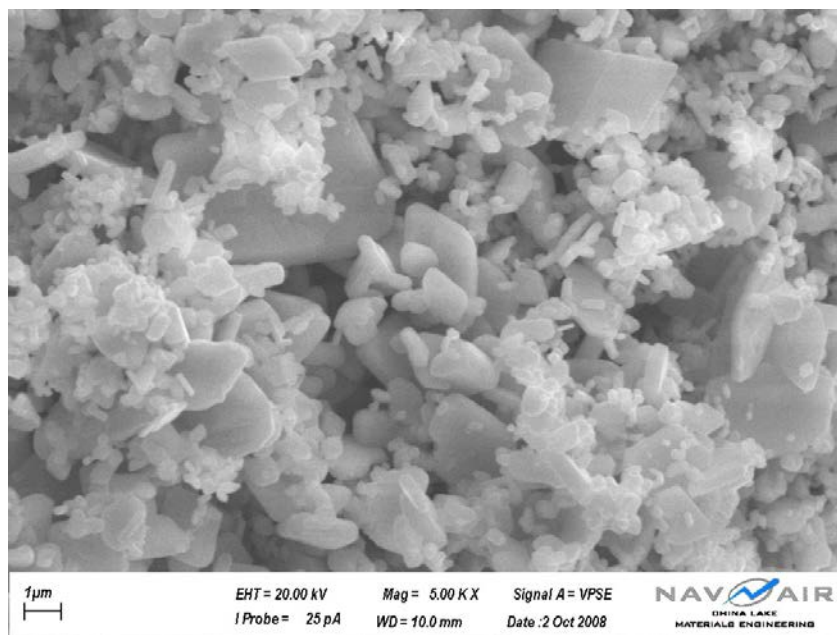
Figure L.18: NAVAIR's Ag_2O

Figure L.19 is the SEM of I_2O_6 made at USC. I_2O_6 is a strong oxidizer that can be used to make very energetic composites with fuels. Unfortunately, the oxidizer proved to be too large to produce high performing energetic nano-composites.
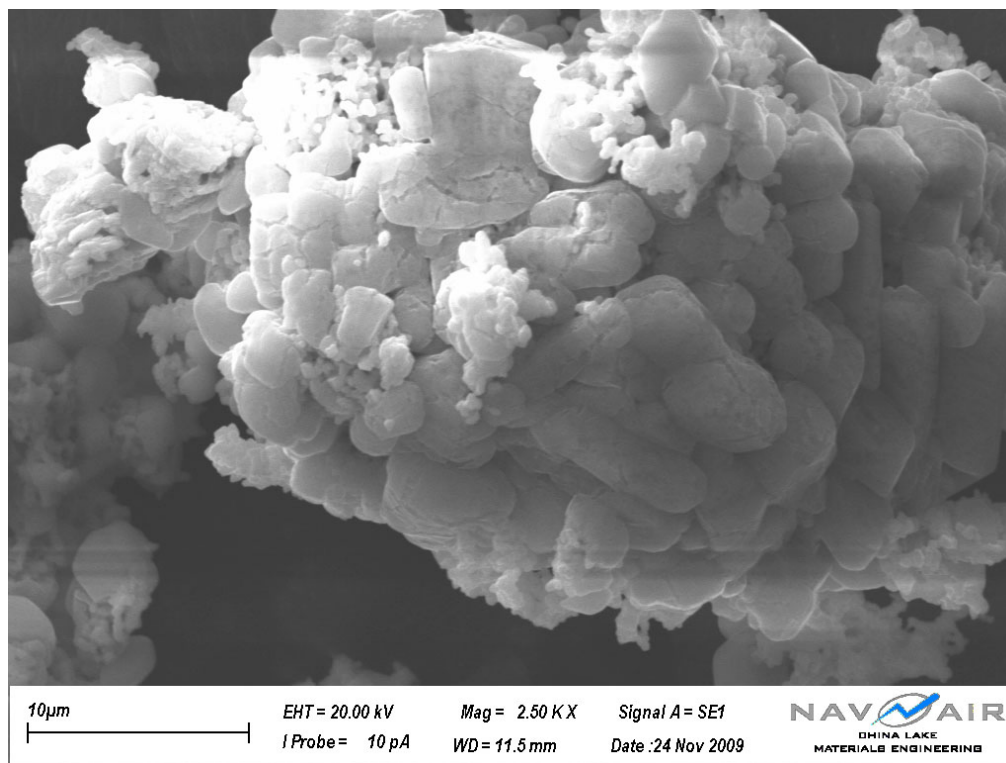
Figure L.19: USC's I_2O_6

Figure L.20 displays the Dyno Nobel low density AN after ball milling. The AN starts off as low density prills with a proprietary stabilizer coating. It is milled as described in Appendix G. Palmitic Acid is used in the milling process as a hydrophobic coating for freshly exposed surfaces. Approximately 0.5 weight percent of Palmitic Acid is placed on the AN.
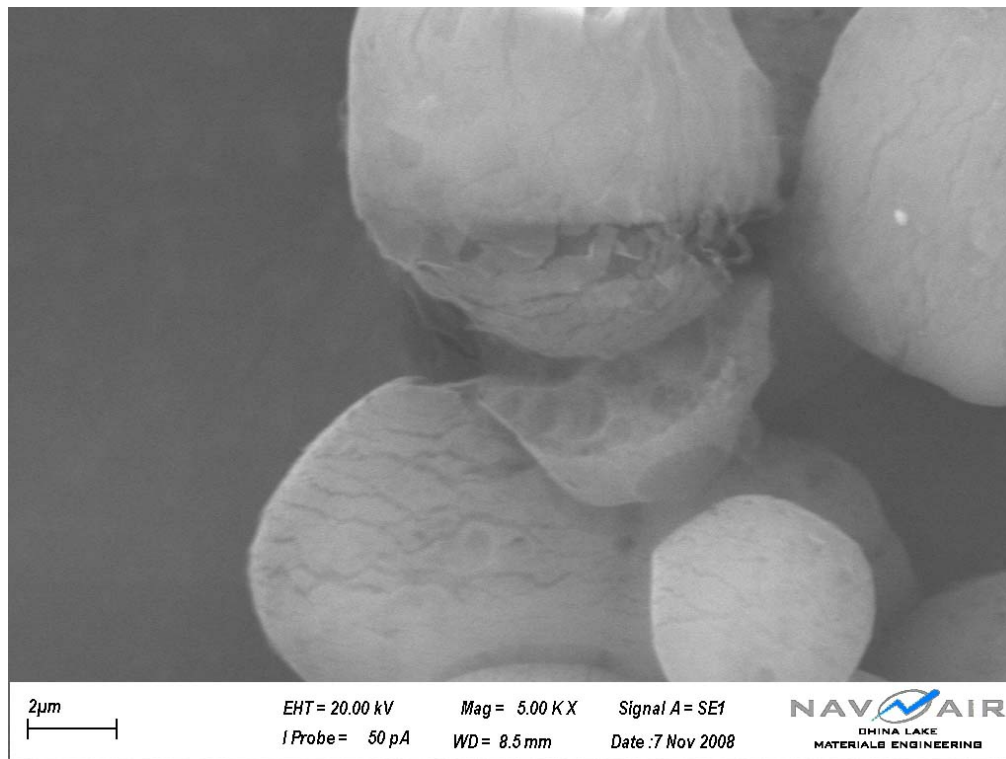
Figure L.20: Porous Low Density AN After Ball Milling

The SEM of the gas generator additive DABT is shown in Figure L.21. The DABT powder is composed of large log shaped crystals. Some of the crystals are more than 100 μm in length. The large particle size may be limiting the gas release rates.
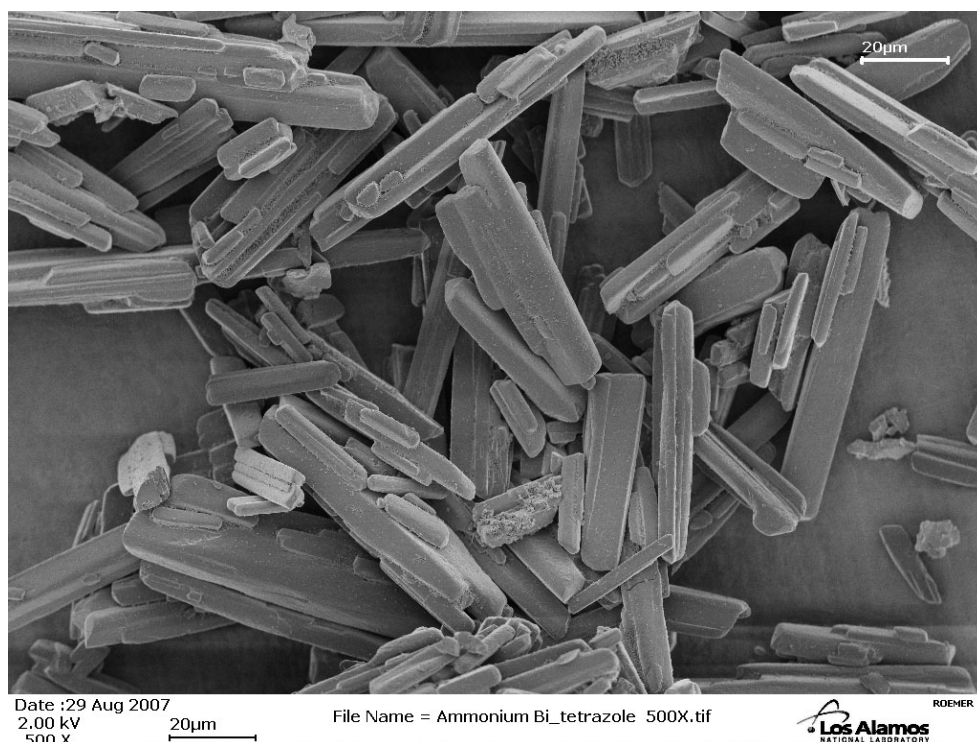
Figure L.21: Diammonium Bitetrazole (DABT)

Figure L.22 is the SEM of the Chevron-Phillips Carbon Black. The BET particle size was 40 nm and appears nearly spherical. The carbon black is critical for electric ignition of primers.
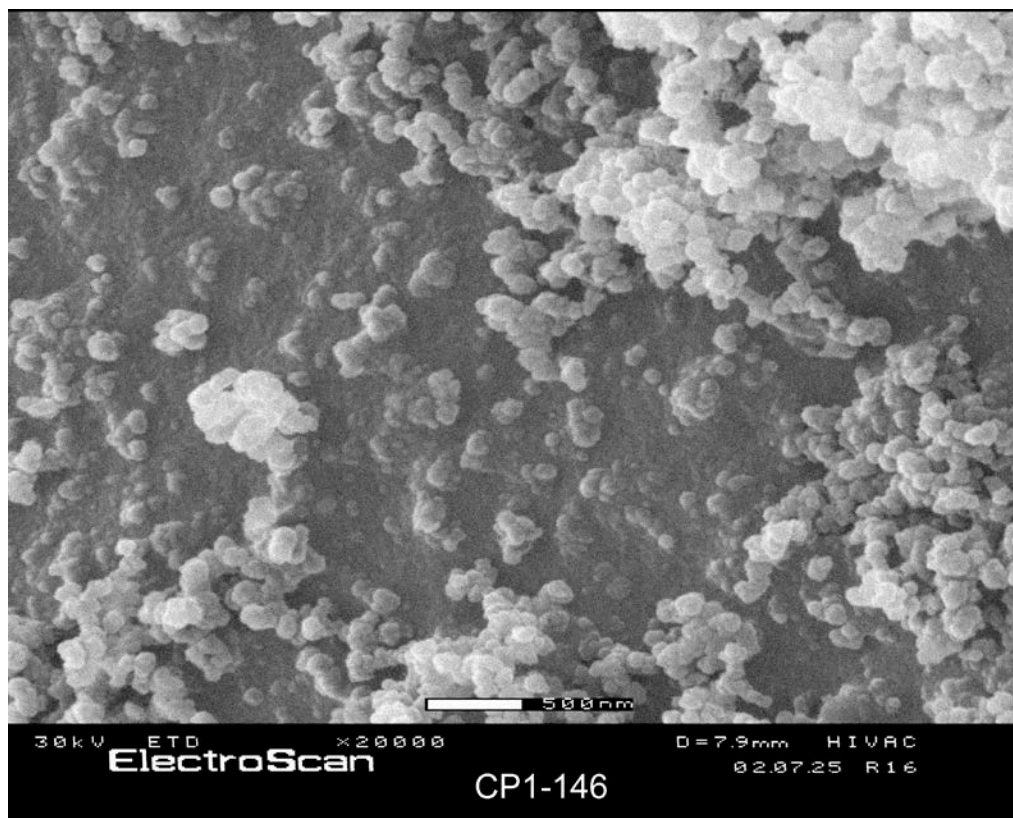
Figure L.22: Chevron-Phillip's Carbon Black

Figure L.23 shows the SEM of a Al(50nm)/MoO$_3$(45nm) sonicated composite. The Al and MoO$_3$ are well mixed and the larger MoO$_3$ orthogonal plates are also coated with nanoparticles.
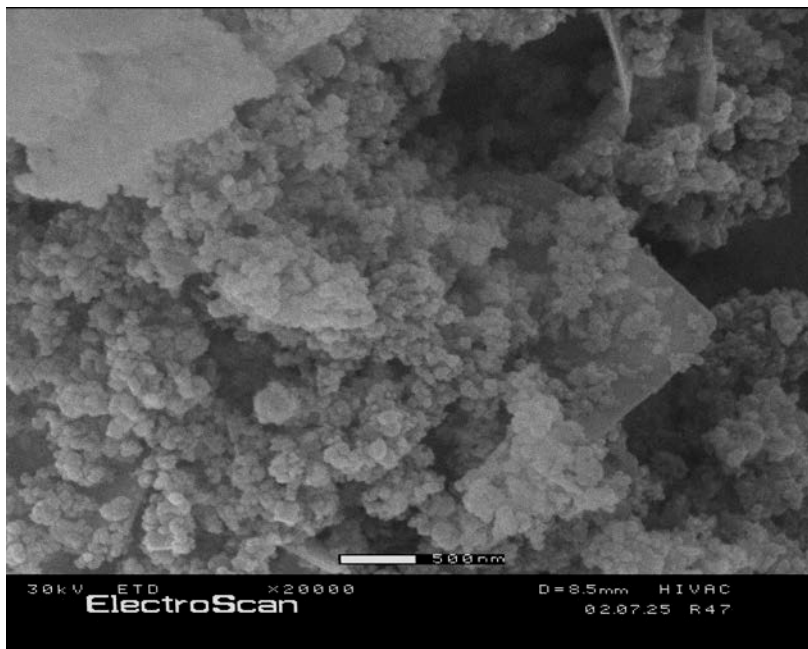
Figure L.23: Al(50nm)/MoO$_3$(45nm)

Figure L.24 is an example of self-assembling spheres. A dry mixture of 80 nm Al and 45 nm MoO$_3$ were dry mixed for 1 minute. Due to high electrostatic interactions, large nearly spherical agglomerates formed. Some of the larger MoO$_3$ plates can be seen as isolated structures. This self-assembling phenomena practically eliminates free nano particles of Al and MoO$_3$ reducing the dust and airborne hazard.
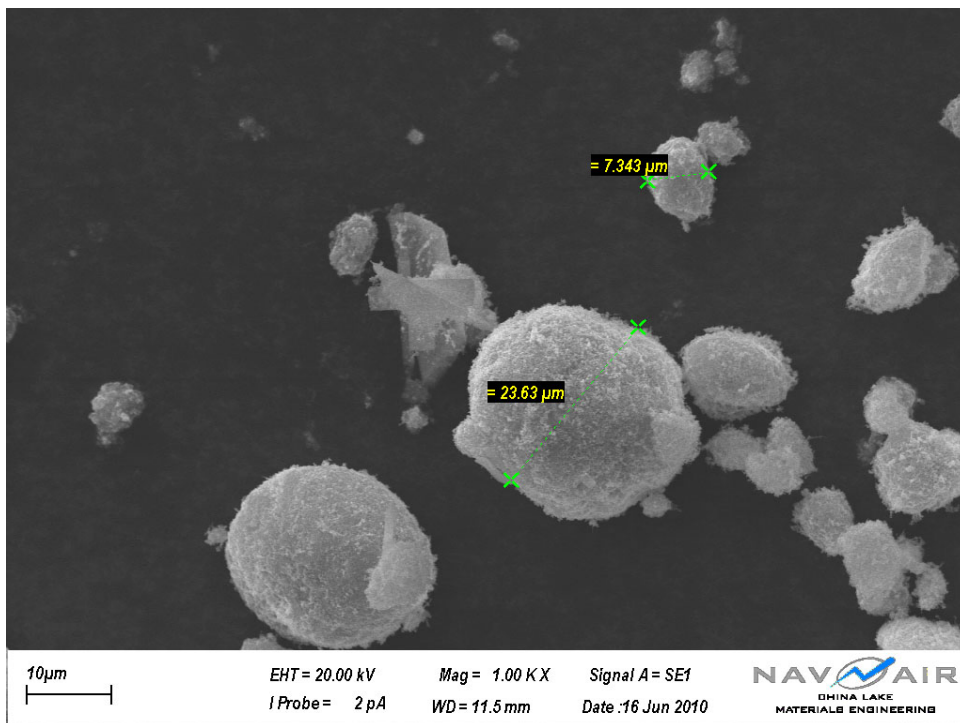
Figure L.24: Self-Assembly of 80 nm Al and 45 nm MoO$_3$ into Large Agglomerates

The agglomerates in Figure L.24 were created by dry mixing the 80 nm Al and 45 nm MoO$_3$ powders in a polyethylene bottle. The contents were shaken in the bottle for 60 seconds. The large micron agglomerates form due to the electrostatic attraction between the 2 solids.

Figure L.25 is the SEM of a Al(80nm)/MoO$_3$ composite prepared by sonication. It is very similar to the SEM of the Al(50nm)/MoO$_3$(45nm) composite. There is a high degree of agglomeration into larger multi-micron structures. These structure explain why the composites do not filter through Whatman 4 filter paper.
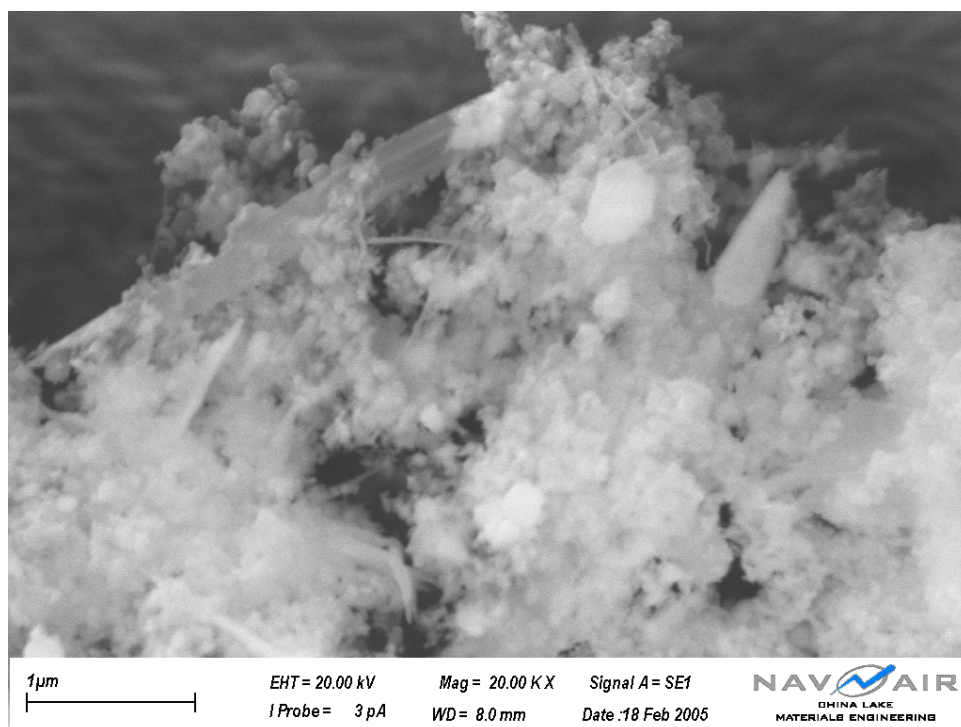
Figure L.25: Al(80nm)/MoO$_3$(45nm) After Sonication

Figure L.26 is the SEM of Al(80nm)/MoO$_3$ prepared by ball milling using Alumina milling balls. The material looks very similar to the sonicated sample in Figure L.25. The MoO$_3$ plates are embedded in the agglomerate.
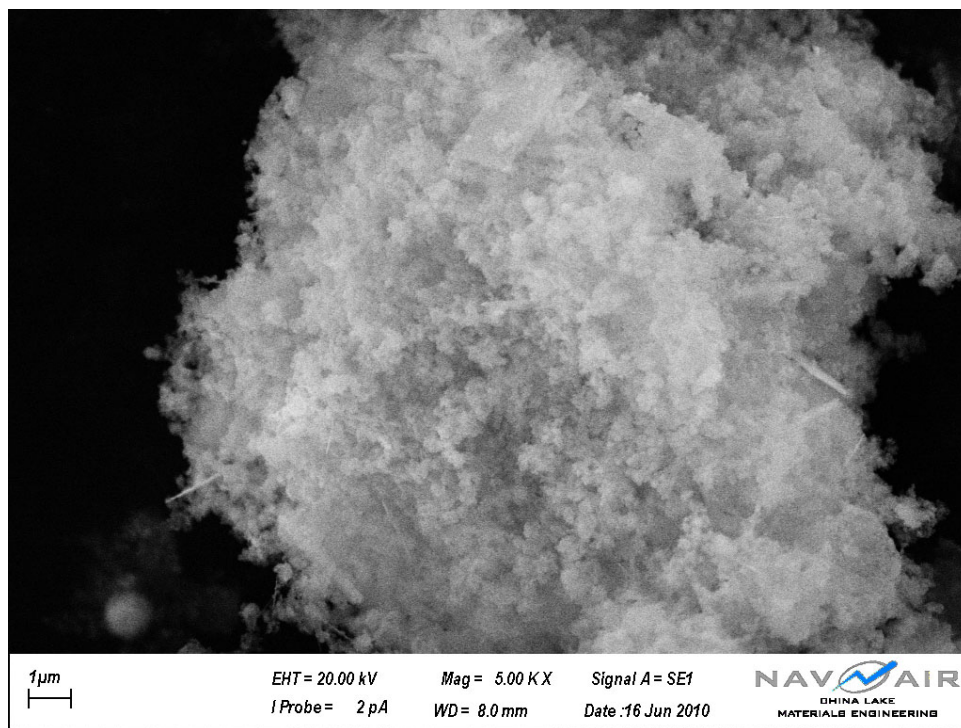
Figure L.26: Al(80nm)/MoO$_3$(45nm) After Ball Milling With Alumina

Figure L.27 is the SEM of Al(80nm)/MoO$_3$(45nm) after ball milling with steel milling balls. The product looks very similar to the products in L.25 and L.26. There isn't a distinguishable difference in appearance of the Al(80nm)/MoO$_3$(45nm) product whether it is made by sonication or ball milling.
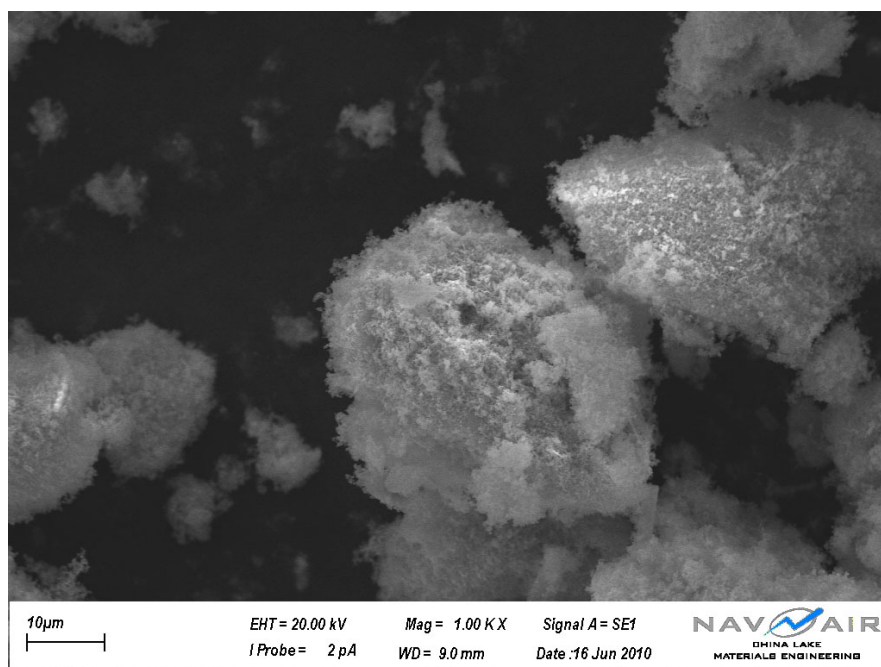
Figure L.27: Al(80nm)/MoO$_3$ After Ball Milling with Stainless Steel

Figure L.28 is the SEM of the Al(80nm)/Bi$_2$O$_3$(10μm) composite made by sonication. Some of the large Bi$_2$O$_3$ logs are embedded in the Al agglomerates but the coating is poor. Free Bi$_2$O$_3$ can be seen and some stick out of the Al agglomerates. There appears to be very little fuel-oxidizer surface contact area. Low performance was obtained from this sample.
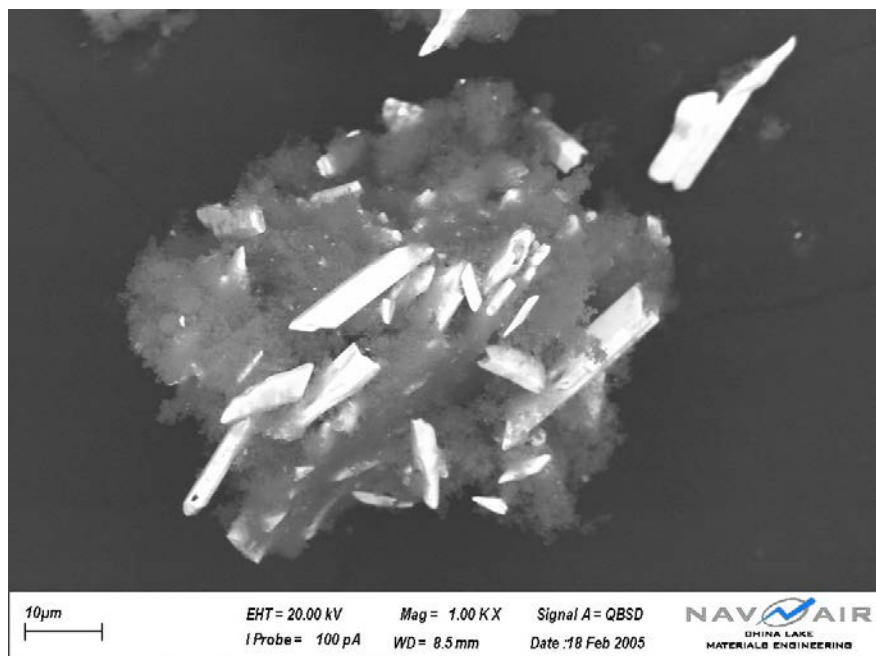
Figure L.28: Al(80nm)/Bi$_2$O$_3$ (10 μm)

Figure L.29 is the SEM of the Al(80nm)/Bi$_2$O$_3$(2.5μm) composite made by sonication. It appears to be very similar to the sample in Figure L.28. There is some Bi$_2$O$_3$ embedded in the Al agglomerate but free Bi$_2$O$_3$ is observed. This sample also gave poor deflection numbers in the Al Pan Dent Test.
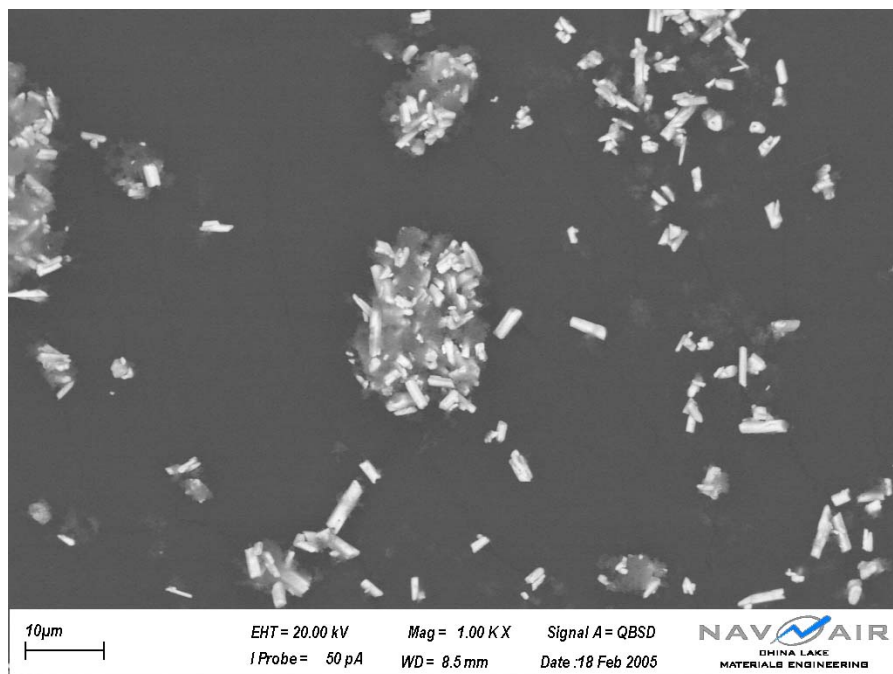
Figure L.29: Al(80nm)/Bi$_2$O$_3$ (2.5 μm)

Figure L.30 is the SEM of the Al(80nm)/Bi$_2$O$_3$(320nm) composite. The composite looks extremely homogenous and appears to be the best mixed sample prepared to date. As expected, this is a high performing ENC.
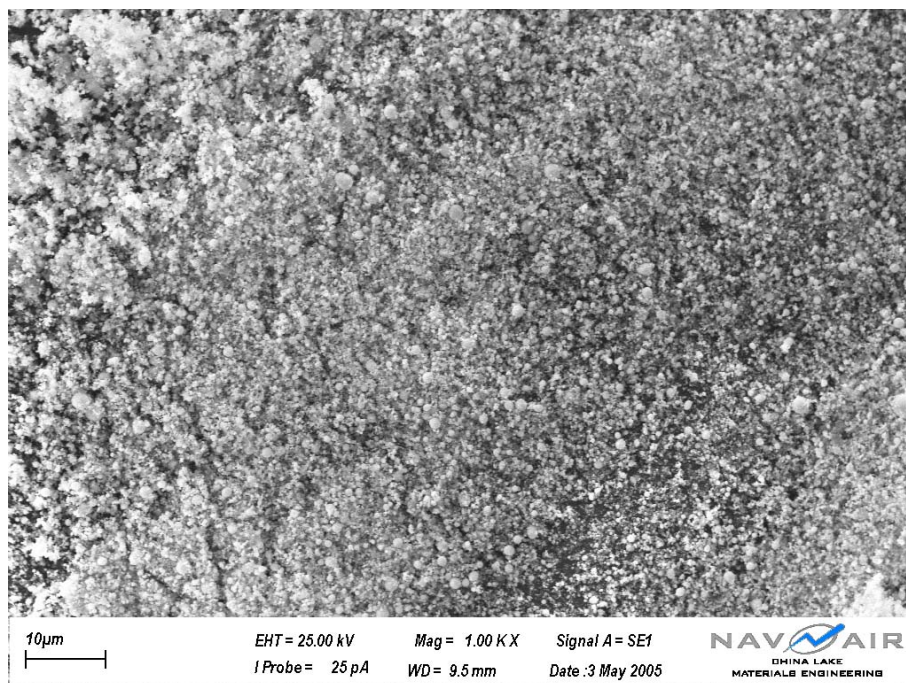
Figure L.30: Al(80nm)/Bi$_2$O$_3$(320nm)

Figure L.31is the SEM of the Al(50nm)/Ag$_2$MoO$_4$ composite made by sonication. The Al is agglomerated over the surface of the larger Ag$_2$MoO$_4$ particles.
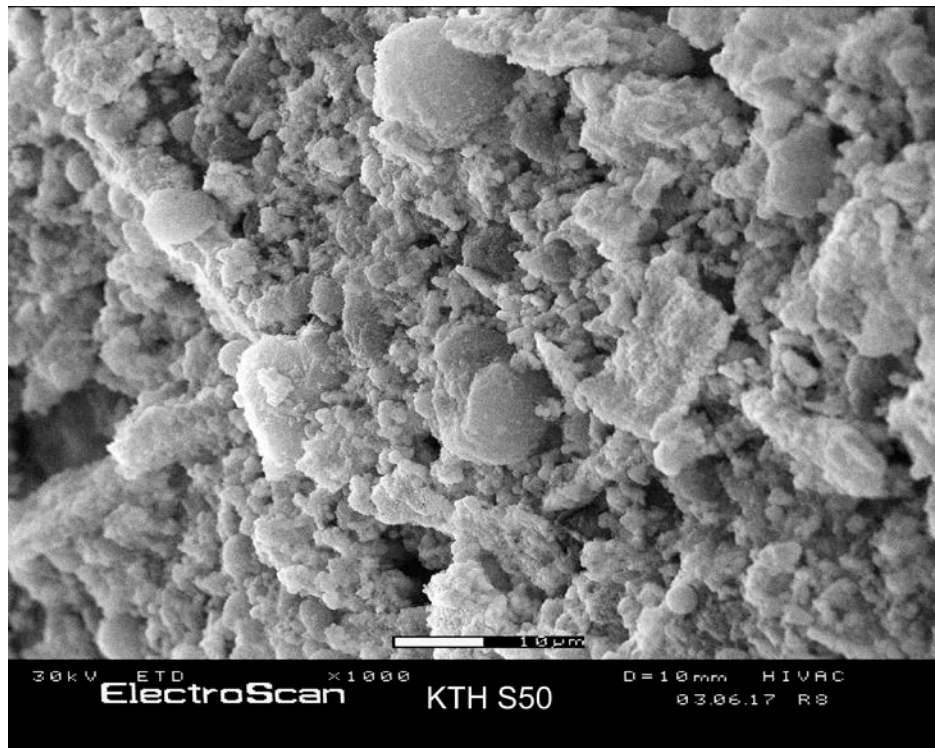
Figure L.31: Al(50nm)/Ag$_2$MoO$_4$

Figure L32 is the SEM of a batch of the Standard LFEP composition. The LFEP is highly agglomerated into large 10 to 100 micron structures.
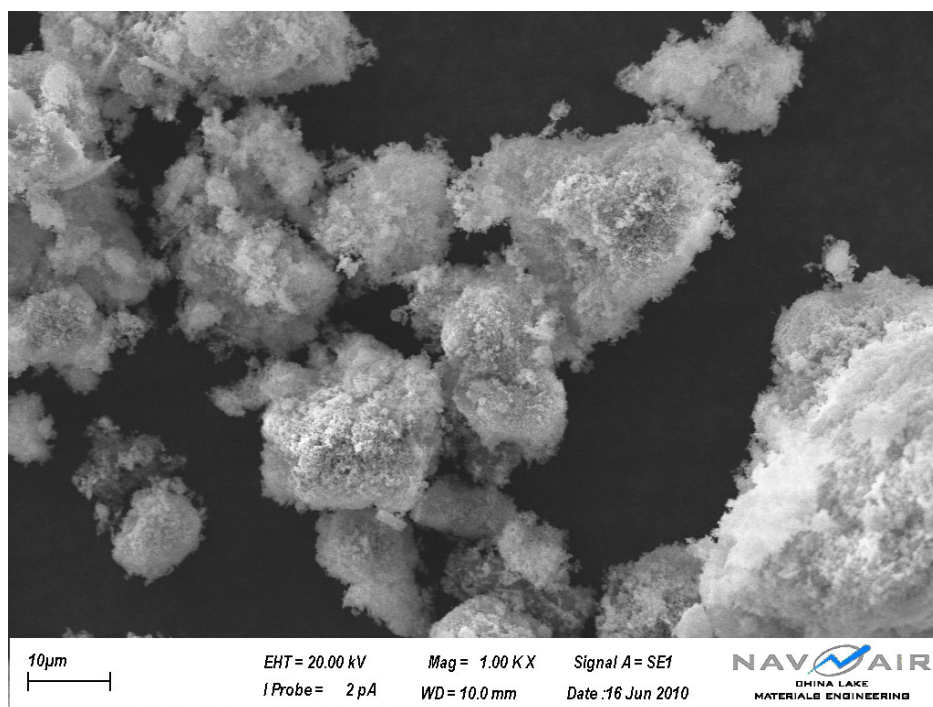
Figure L.32: Al(80nm)/MoO$_3$/AN/Kel-F/C (Standard LFEP)

Figure L.33 is the SEM of the product from the Al(80nm)/MoO$_3$ ENC ignition. The 3 micron sphere is made up of mainly alumina with nanomaterials fused to the surface.
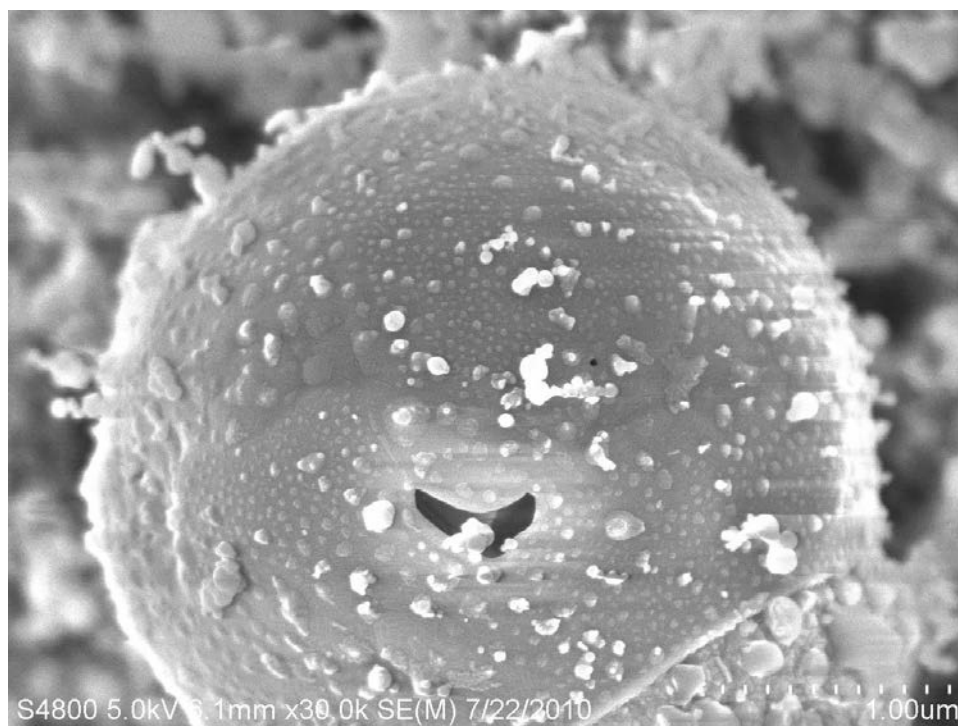
Figure L.33: Micron Al$_2$O$_3$ Sphere From Al(80nm)/MoO$_3$ ENC Ignition

Figure L.34 is the combustion product from the Al(80nm)/Ag$_2$O ENC. This composite produces less gas and larger alumina spheres.
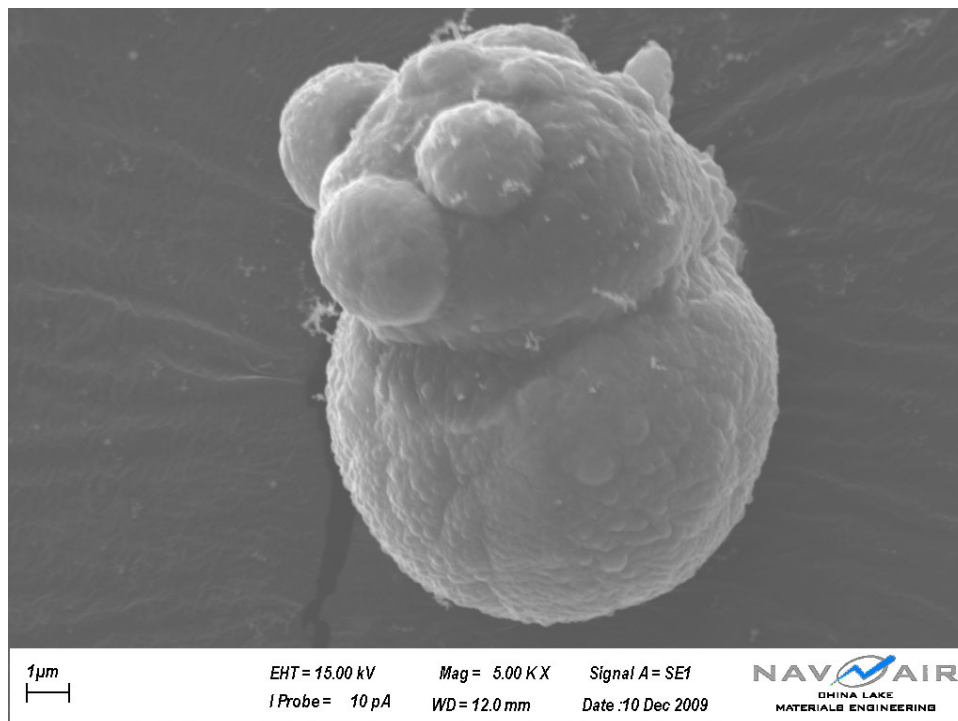
Figure L.34: Micron Al$_2$O$_3$ Spheres From Al(80nm)/Ag$_2$O ENC Ignition

Figure L.35 is the SEM of the products from the ignition from the Al(80nm)/AgIO$_3$ ENC. The alumina spheres range in size from1 to 5 microns and AgI nanoparticles decorate the surface. Free submicron AgI particles are also evident.
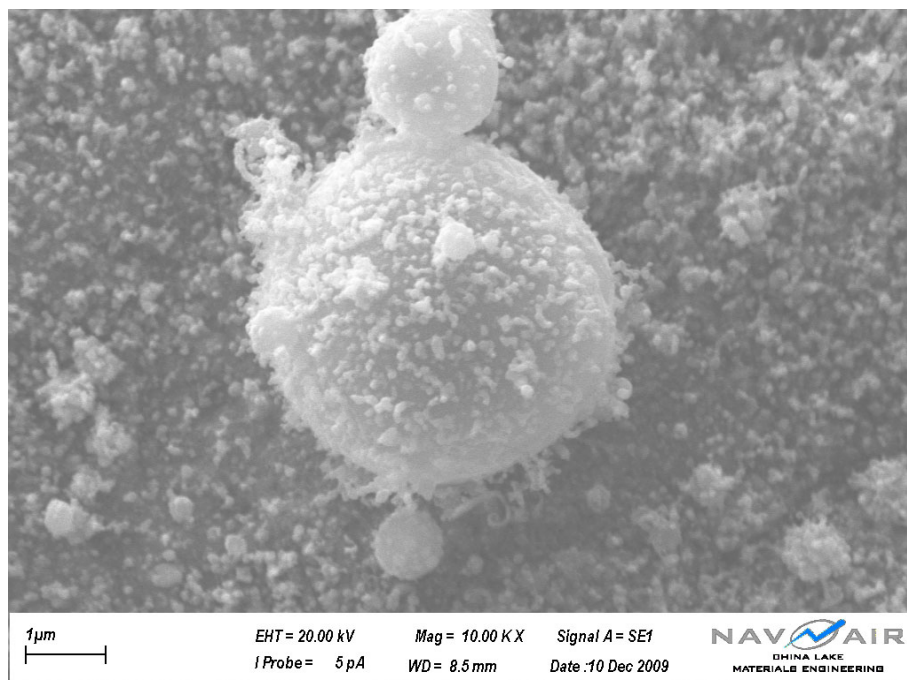
Figure L.35: Micron Al_2O_3 Spheres From Al(80nm)/AgIO$_3$ ENC Ignition

Figure L.36 is the combustion product from the Al(80nm)/WO$_3$ ENC. Once again alumina spheres are observed in the range of 1 to 10 microns. The surfaces are decorated with submicron particles.
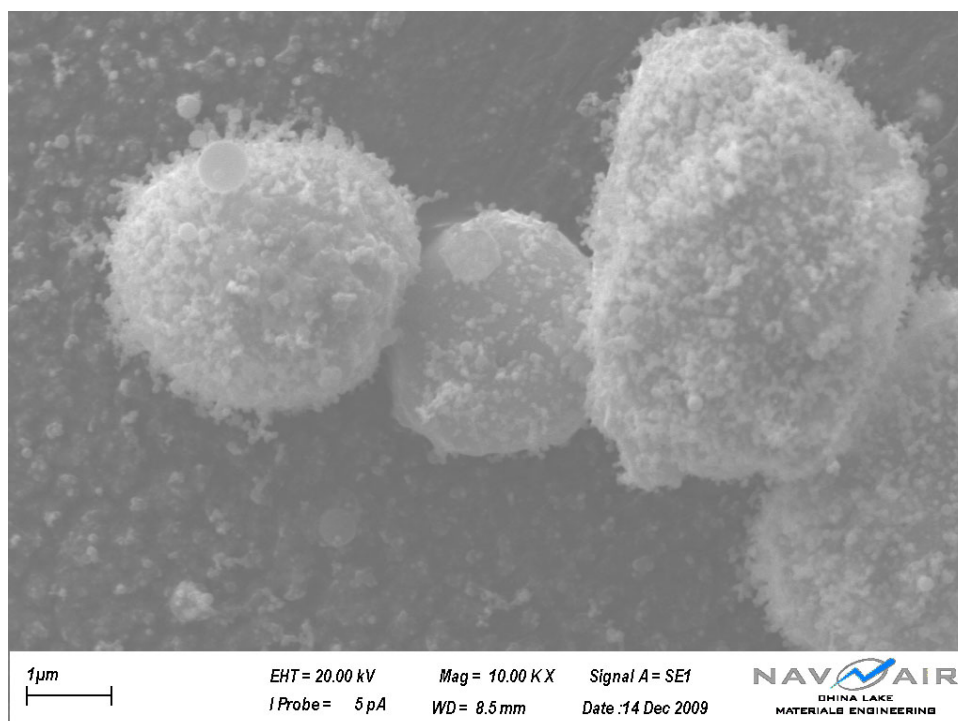
Figure L.36: Micron Al_2O_3 Spheres From Al(80nm)/WO_3 ENC Ignition

Figure L.37 is the combustion product from the Al(80nm)/Ag_2MoO_4 ENC. Once again large spherical alumina balls form and in this case, several are fused together. The surface is again decorated with submicron particles.
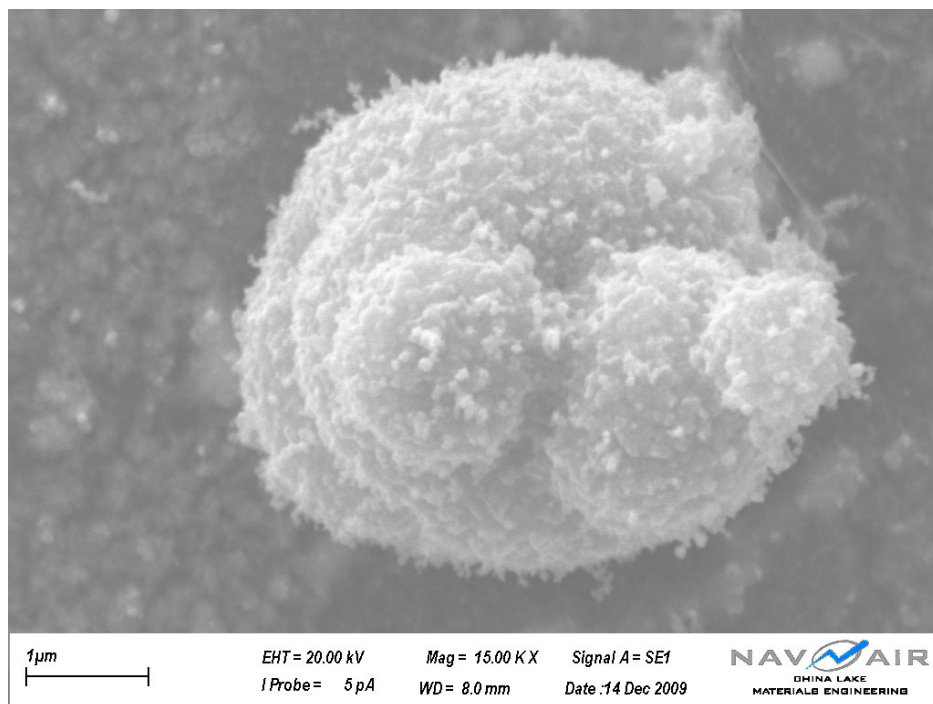
Figure L.37: Micron Al_2O_3 Spheres From Al(80nm)/Ag_2MoO_4 ENC Ignition

In Figure L.38, the combustion products from the Al(80nm)/$Bi(IO_3)_3$ ENC are large spherical alumina balls. However, there are also a lot of free nano particles. This ENC produces more gas byproducts than most ENCs and this may lead to a larger quantity of free nanoproducts.
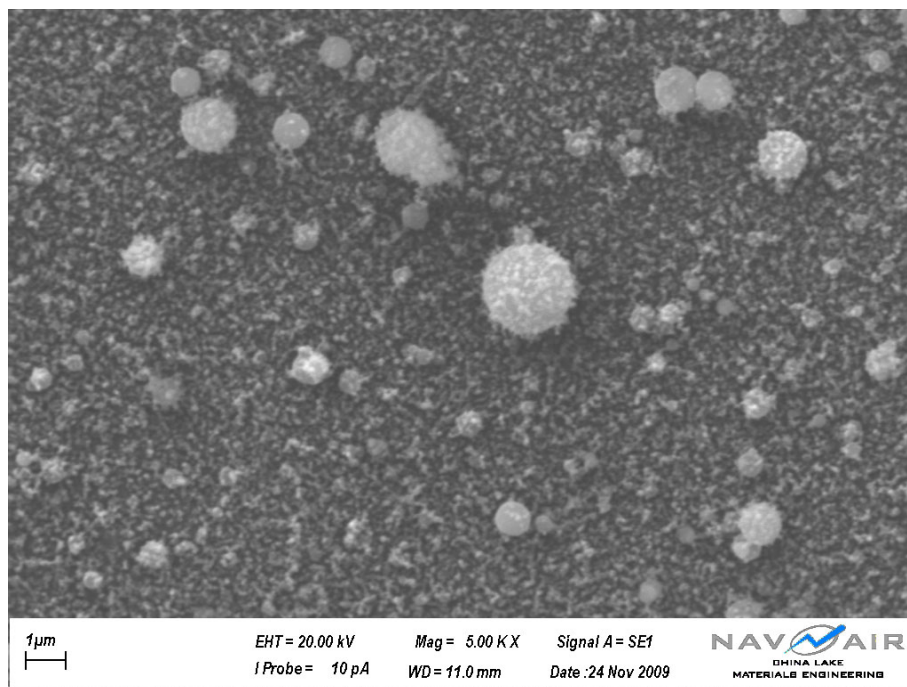
Figure L.38: Micron Al_2O_3 Spheres From Al(80nm)/Bi(IO$_3$)$_3$ ENC Ignition

Figure L.39 shows the combustion products from the Al(80nm)/CuO ENC. Large spherical alumina balls are present but very little nanoparticles are present. Some submicron products are fused to the surface of the large alumina spheres.
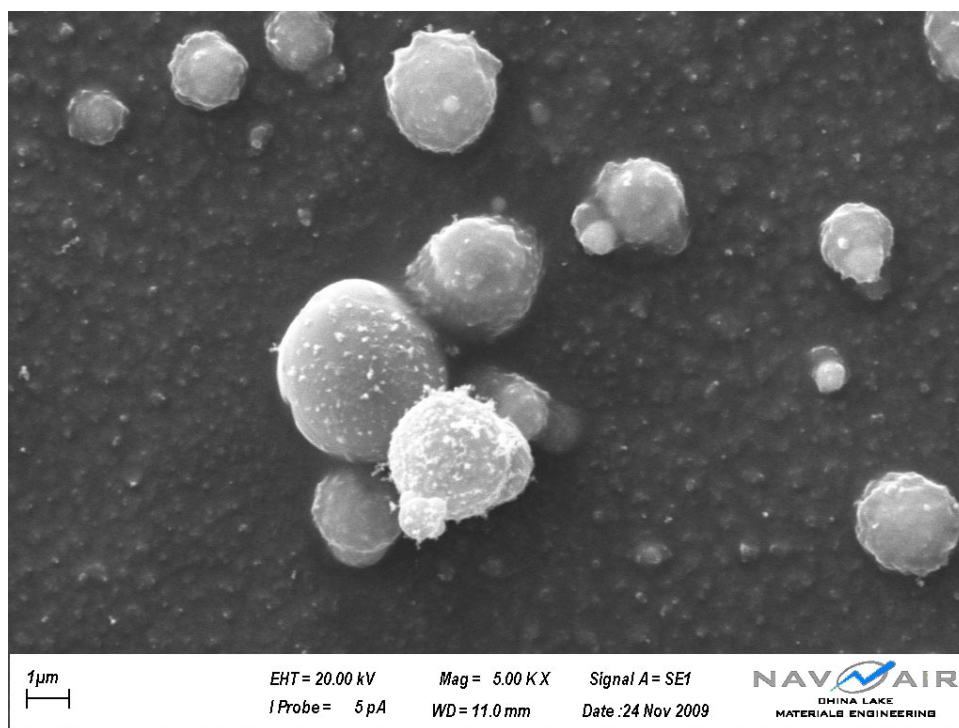
Figure L.39: Micron Al_2O_3 Spheres From Al(80nm)/CuO ENC Ignition

Figure L.40 shows the products from the Ti(nano)/AgIO$_3$ ENC. The TiO$_2$ spheres are similar to the alumina spheres and the surfaces are coated with AgI particles.
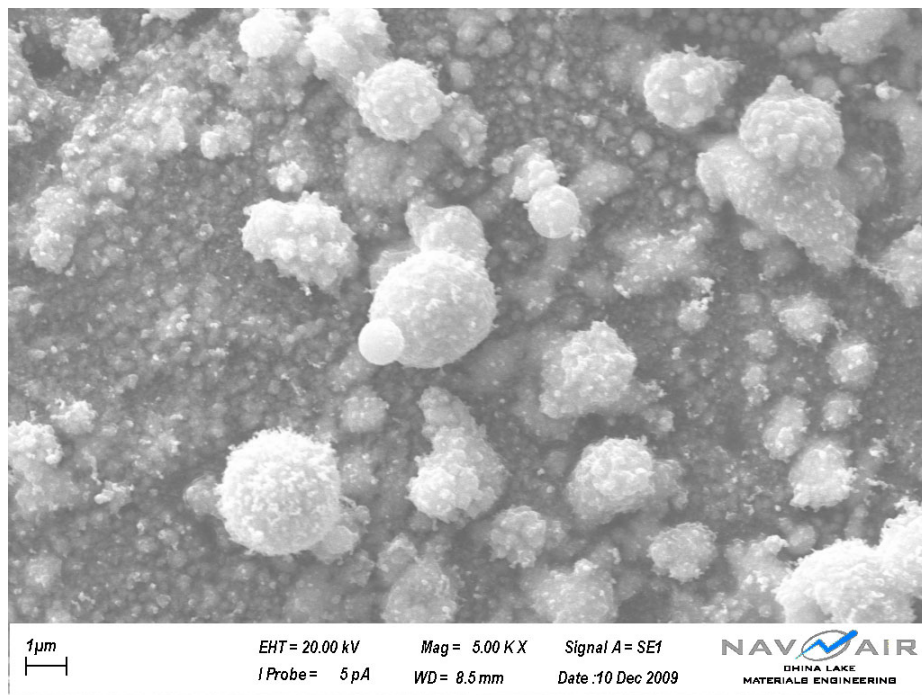
Figure L.40: Micron TiO$_2$ and AgI Spheres From Ti(nano)/AgIO$_3$ ENC Ignition

Figure L.41 shows the products from a Standard LFEP batch. The majority of the products are large spherical alumina balls with small particles fused to the surface.
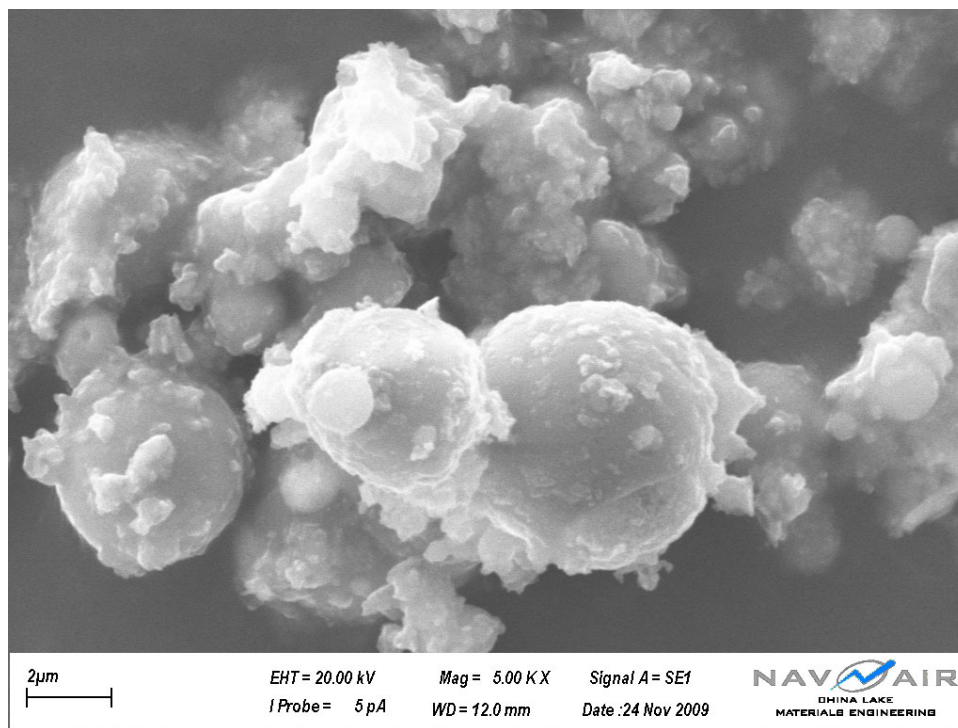
Figure L.41: Micron Products From Al(80nm)/MoO$_3$/AN/Kel-F/C Ignition

Appendix M: Deflections For LFEP Precursors and Primers With AUR-ATs

Appendix M contains the Al Pan Deflection data for LFEP during the various synthetic stages and the final AUR-ATs for the batch. Typically, the ENC Al/MoO$_3$ is made first, then AN was added to form the Al/MoO$_3$/AN intermediate, then Kel-F added, and finally carbon added. The KTHZ60 formulation was selected as the standard LFEP formulation and is highlighted in green. Other formulation with similar compositions resulted in higher AUR-ATs or no fire. The reason for the poorer performance is highlighted in yellow.

		Al/MoO$_3$		Al/MoO$_3$/AN		Al/MoO$_3$/AN/Kel-F		Al/MoO$_3$/AN/Kel-F/C		Scale g	AUR-AT	Notes
	Al (Size)	Molar Ratio	Deflection	Wt% AN	Deflection	Wt% Kel-F	Deflection	Wt% C	Deflection			
KTHY62	Al(80nm) M2210	2.63	NA	38.5%	NA	2%	NA	2%	NA	3	3.05	Sonic
KTHY64	Al(80nm) M2210	2.63	NA	30.0%	NA	2%	NA	2%	NA	2	3.04	Sonic
KTHZ50	Al(80nm) M2210	2.50	149	31.4%	467	2%	466	2%	361	5	3.33	Sonic
KTHZ60	Al(80nm) M2210	2.65	143	30.5%	NA	2%	432	2%	402	5	3.03	Sonic
KTHZ60	Al(80nm) M2210	2.65	143	30.5%	NA	2%	432	2%	402	5	3.01	Sonic
KTHZ60	Al(80nm) M2210	2.65	143	30.5%	NA	2%	432	2%	402	5	3.02	Sonic
KTHZ63	Al(80nm) M2210	2.63	NA	30.0%	NA	2%	NA	2%	527	8	Picatinny	
KTHZ64	Al(80nm) M2210	2.65	NA	29.2%	NA	3.4%	NA	2%	358	6	3.25	Alt Syn
KTHAB4	Al(80nm) M2453			30.1%		Al/MoO$_3$/AN/Teflon (2%)			292	6	No Fire	Teflon
KTHAB26	Al(Alex)	3.04	158 (72h)	30.4%		2%	NA	2%	NA	5	3.59	
KTHAB66	Al(Alex)	2.91	166 (39h)	Al/MoO$_3$ + 0.67Al(80)/AN (31.4%)		2.0%	NA	2%	NA	5	3.67/4.88	
KTHAB67	Al(Alex)	2.91	166 (39h)	Al/MoO$_3$ + 2Al(80)/AN (31.4%)		2%	NA	2%	NA	5	3.64	

Sample	Material											Notes
KTHAB95	Al(80nm) M2210	2.65	NA	28.8%	NA	2%	NA	2%	NA	5	No Fire	Wet C
KTHAD03	Al(Alex)	2.68	160(24h)	37.0%	414	2%	161	2%	NA	5	117	
KTHAD04	Al(Alex)	2.68	160(24h)	37.0%	414	2%	161	2% Graphite	NA	1	249	
KTHAD05	Al(Alex)	2.68	160(24h)	37.0%	414	2%	161	2% Zn Dust	NA	1	239	
KTHAD06	Al(Alex)	2.68	160(24h)	37.0%	414	2%	161	2% Ag Flake	NA	1	No Fire	
KTHAD13	Al(Alex)/ MoO$_3$/Bi$_2$O$_3$		424	30.0%	NA	2%	NA	2%	0	5	3.86/7.88	
KTHAD31B	Al(80nm) M2210	2.65	105(48h)	38.8%	NA	2%	503	2%	NA	5	15.07	BM/New Dies Set
KTHAD42A	Al(80nm) M2210	2.65	160(62h)	33.0%	739	2%	637	2%	NA	5	51.57	BM/New Dies Set
KTHAD54	Al(80nm) M2210	2.62	135	30.0%	NA	2%	587	2%	NA	5	3.63	
KTHAD56	Al(80nm) M2210	2.65	160(62h)	31.2%	NA	2%	751	2%	NA	5	3.53	BM
KTHAD60B	Al(80nm) M2210	2.65	160(62h)	30.0%	NA	2%	570	2%	446	4	3.21	BM
KTHAF37B	Al(80nm) M2671	2.67	216(37h-s)	29.6%	868	2%	750	0%		136	86	Scale-Up Sample
KTHAF40	Al(80nm) M2671	2.67	212(24h-s)	29.6%	607	2%	637	0%		148		Scale-Up Sample
KTHAF42	Al(80nm) M2671	2.63	219(96h)	29.4%	NA	2%	577	0%		102		Scale-Up Sample
KTHAF80	Al(80nm) M2671	2.67	212(72h)	33.5%	NA	2%	637	2%	NA	22	3.93	BM
KTHAF144	Al(80nm) M2210	2.69	212(96h)	30.0%	NA	2%	328	2%	360	109	3.23	BM, Scale-up Carbon mixing

APPENDIX N: All-Up Round Action Time Data

STANDARD LFEP PREPARED BY WET LOADING, DRYING, AND PRESSING WITH OLD DIE

Formulation	Date Loaded	Date Tested	Ambient Temperature	Conditioned Temperature	Estimated Primer Temperature	Action Time	Pressure	Velocity
			°F	°F	°F	ms	ksi	fps
KTHZ60	11/20/2007	11/28/2007	60	Ambient	72	2.98	50.19	3320
KTHZ60	11/20/2007	11/28/2007	60	Ambient	72	2.95	50.57	3299
KTHZ60	11/20/2007	11/28/2007	60	Ambient	72	3.06	52.85	3308
KTHZ60	11/20/2007	11/28/2007	60	Ambient	72	3.09	49.05	3305
KTHZ60	11/20/2007	11/28/2007	60	Ambient	72	3.09	49.43	3325
KTHZ60	11/20/2007	11/28/2007	60	Ambient	72	3.05	51.33	3315
KTHZ60	11/20/2007	11/28/2007	60	Ambient	72	3.12	47.68	3330
KTHZ60	11/20/2007	11/28/2007	60	Ambient	72	2.85	55.51	3335
KTHZ60	11/20/2007	11/28/2007	60	Ambient	72	3.03	49.81	3317
KTHZ60	11/20/2007	11/28/2007	60	Ambient	72	3.04	50.95	3322
KTHZ60	11/20/2007	11/28/2007	60	Ambient	72	3.02	47.43	3306
KTHZ60	11/20/2007	11/28/2007	60	Ambient	72	2.9	51.71	3328
KTHZ60	11/20/2007	11/28/2007	60	Ambient	72	3	50.57	3318
KTHZ60	11/20/2007	11/28/2007	60	Ambient	72	2.98	50.95	3315
KTHZ60	11/20/2007	11/28/2007	60	Ambient	72	2.99	49.33	3321
KTHZ60	11/20/2007	11/28/2007	60	Ambient	72	3.09	49.05	3310
KTHZ60	11/20/2007	11/28/2007	60	Ambient	72	3.06	49.81	3323
KTHZ60	11/20/2007	11/28/2007	60	Ambient	72	3.03	50.57	3315
KTHZ60	11/20/2007	11/28/2007	60	Ambient	72	3.13	51.71	3316
KTHZ60	11/20/2007	11/28/2007	60	Ambient	72	3.08	49.05	3319
KTHZ60	11/20/2007	11/28/2007	60	Ambient	72	2.98	50.19	3302
KTHZ60	11/20/2007	11/28/2007	60	Ambient	72	3.05	48.67	3308
KTHZ60	11/20/2007	11/28/2007	60	Ambient	72	3.1	52.47	3301
KTHZ60	11/20/2007	11/28/2007	60	Ambient	72	2.98	50.19	3303

KTHZ60	11/20/2007	11/28/2007	60	Ambient	72	2.89	53.61	3315
KTHZ60	11/20/2007	11/28/2007	60	Ambient	72	3.06	49.43	3306
KTHZ60	11/20/2007	11/28/2007	60	Ambient	72	3.05	50.95	3298
KTHZ60	11/20/2007	11/28/2007	60	Ambient	72	3.03	49.81	3289
KTHZ60	11/20/2007	11/28/2007	60	Ambient	72	3.01	49.05	3295
KTHZ60	11/20/2007	11/28/2007	60	Ambient	72	3.02	50.19	3309
KTHY64		4/12/2007	60	Ambient	70	3.07	47.2	3281
KTHY64		4/12/2007	60	Ambient	70	3.02	48	3332
KTHY64		4/12/2007	60	Ambient	70	3.09	49.8	3325
KTHY64		4/12/2007	60	Ambient	70	3.09	47.2	3316
KTHY64		4/12/2007	60	Ambient	70	3.07	47.5	3287
KTHY64		4/12/2007	60	Ambient	70	2.91	48.3	3302
KTHY62		4/12/2007	60	Ambient	70	3	50.5	3329
KTHY62		4/12/2007	60	Ambient	70	3.15	49.1	3311
KTHY62		4/12/2007	60	Ambient	70	2.98	49.8	3354
KTHY62		4/12/2007	60	Ambient	70	3.04	48.4	3326
KTHY62		4/12/2007	60	Ambient	70	3.1	48	3334
KTHY50A		4/12/2007	60	Ambient	70	3.05	47.6	3313
KTHY50A		4/12/2007	60	Ambient	70	3.08	47.9	3318
KTHY50A		4/12/2007	60	Ambient	70	3.14	48.4	3329
KTHY50A		4/12/2007	60	Ambient	70	3.07	47.9	3316
KTHY50A		4/12/2007	60	Ambient	70	3.03	48	3315
					Average	3.03	49.69	3314.37
					Std Deviation	0.07		

Formulation	Date Tested	Ambient Temperature	Conditioned Temperature	Estimated Primer Temperature	Action Time	Pressure	Velocity
KTHY64	4/13/2007	60	-65	-74	3.09	53.2	3184
KTHY64	4/13/2007	60	-65	-63	3.27	50.9	3182
KTHY64	4/13/2007	60	-65	-59	3.16	48	3185
KTHY62	4/13/2007	60	-65	-65	3.13	48.7	3187
KTHY62	4/13/2007	60	-65	-72	3.27	50.9	3204
KTHY62	4/13/2007	60	-65	-61	3.05	52.5	3177
KTHY62	4/13/2007	60	-65	-61	3.06	48.8	3207
KTHY62	4/13/2007	60	-65	-75	3.1	49.1	3217
				Average	3.14	50.3	3193
				STD Deviation	0.09		

STANDARD FORMULATION WITH Al/MoO$_3$ of 2.62 to 2.70

STANDARD LFEP FORMULATION (66% Al/MoO$_3$, 30% AN, 2% Kel-F, 2% Carbon)

BATCH KTHAF144	Al/MoO$_3$ Molar Ratio	Wt% Al/MoO$_3$	Wt% AN	Wt% Kel-F	Wt% C					
	2.69	67%	29%	2%	2%					
B52 BALLISTIC TEST LABORATORY										
Code 478200D										
Date: 20 June 2011				Test Engineer: D. Herigstad					Instrumentation: L. Wheeler	
Test: Green Primer Trials (Corrected)				Firing Officer: C. Lancaster					Gunner: A. Camacho	
Event No.	Time	Peak Pressure (kpsi)	Velocity (fps)	Action Time (ms)	Prop. Wt. (grs)	Proj. Wt. (grs)	Primer Type	Barrel Temp (deg F)	Round Number	Notes
1	15:17								Checkout	No Data 3 Rounds expended
2	8:53	36.12	3492	2.7	580.0				chkout 1	21-Jun-11

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3	8:59	38.95	3471	2.70	580.0					chkout 2	
4	9:08	44.22	3527	3.15	580.0					1W	No Fire. Fired on second attempt @ 14:50
5	9:17	41.84	3468	3.38	580.0					2W	
6	9:19	39.37	3495	3.26	580.0					3W	
7	10:26	40.04	3470	3.37	580.0					4W	
8	11:14	43.88	3650	2.9	580.0					5W	
9	13:56	41.84	3458	3.24	580.0					6W	
10	13:59	43.65	3491	3.12	580.0					7W	
11	14:02	40.76	3458	3.43	580.0					8W	
12	14:04	41.14	3453	3.27	580.0					9W	
13	14:08	38.40	3405	3.30	580.0					10W	
14	14:10	36.43	3436	3.37	580.0					11W	
15	14:13	43.20	3542	3.31	580.0					12W	
16	14:16	43.65	3473	3.08	580.0					13W	
17	14:19	39.54	3482	3.06	580.0					14W	
18	14:21	43.29	3437	3.05	580.0					15W	
19	14:24	43.29	3443	3.35	580.0					16W	
20	14:27	48.67	3428	3.27	580.0					17W	
21	14:29	42.21	3470	3.15	580.0					18W	
22	14:31	39.68	3461	3.15	580.0					19W	
23	14:35	41.84	3467	3.17	580.0					20W	
24	14:37	42.21	3477	3.18	580.0					21W	
25	14:39	38.95	3484	3.24	580.0					22W	
26	14:42	40.04	3511	3.14	580.0					23W	
27	14:44	44.22	3496	3.23	580.0					24W	
28	14:47	45.1	3513	3.17	580.0					25W	
			3480	3.21	AVE						
			47	0.14	STD						
AMMUNITION LOT NUMBER: KTHAF144 Dry				OAT 95 deg F							
Propellant: WC 868											
Projectiles: PGU-27/B											
Loaded By: L. Wheeler											
Barrel Serial Number: 026											
Breech Block Serial Number: 008											

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B52 BALLISTIC TEST LABORATORY
Code 478200D

Date: 20 June 2011					Test Engineer: D. Herigstad					Instrumentation: L. Wheeler
Test: Green Primer Trials (Corrected)					Firing Officer: C. Lancaster					Gunner: A. Camacho
Event No.	Time	Peak Pressure (kpsi)	Velocity (fps)	Action Time (ms)	Prop. Wt. (grs)	Proj. Wt. (grs)	Primer Type	Barrel Temp (deg F)	Round Number	Notes
1	11:17	43.88	3647	3.21	580.0				1D	
2	11:21	50.72	3451	3.52	580.0				2D	
3	11:25	46.77	3467	3.40	580.0				3D	
4	11:27	42.21	3462	3.25	580.0				4D	
5	11:30	43.54	3476	3.25	580.0				5D	
6	15:10	45.25	3494	3.19	580.0				6D	
7	15:13	46.77	3486	3.17	580.0				7D	
8	15:15	43.88	3494	3.27	580.0				8D	
9	15:18	44.56	3498	3.69	580.0				9D	
10	15:20	46.54	3502	3.14	580.0				10D	
11	15:23	45.25	3506	3.16	580.0				11D	
12	15:25	45.82	3509	3.33	580.0				12D	
13	15:28	42.17	3504	3.18	580.0				13D	
14	15:30	48.35	3526	3.24	580.0				14D	
15	15:32	45.63	3493	3.30	580.0				15D	
16	15:35	44.01	3484	3.30	580.0				16D	
17	15:38	44.90	3466	3.22	580.0				17D	
18	15:41	46.90	3467	3.14	580.0				18D	
19	15:43	43.29	3486	3.20	580.0				19D	
20	15:46	49.07	3489	3.16	580.0				20D	
21	15:48	44.01	3502	3.12	580.0				21D	
22	15:51				580.0				22D	NO DATA
23	15:53	47.26	3454	3.15	580.0				23D	
24	15:56	44.90	3483	3.18	580.0				24D	
25	15:58				580.0				25D	

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			3493	3.25	AVE					
			39	0.14	STDDEV					
AMMUNITION LOT NUMBER: KTHAF144 Dry					OAT 95 deg F					
Propellant: WC 868										
Projectiles: PGU-27/B										
Loaded By: L. Wheeler										
Barrel Serial Number: 026										
Breech Block Serial Number: 008										

Test No.	BATCH KTHZ60 Formulation Fluorinert	Date Rec mm/dd/yy	Date Loaded mm/dd/yy	Date Tested mm/dd/yy	Ambient Temperature °F	Conditioned Temperature °F	Estimated Primer Temperature °F	Action Time ms	Pressure ksi	Velocity fps	Notes
1	M52A3B1			11/28/07	60	Ambient	72	2.88	45.25	3255	Checkout 1
24	KTHZ60		11/20/07	11/28/07	60	Ambient	72	2.93	48.67	3312	7500psi Hexane
25	KTHZ60		11/20/07	11/28/07	60	Ambient	72	2.98	50.19	3320	7500psi Hexane
26	KTHZ60		11/20/07	11/28/07	60	Ambient	72	2.95	50.57	3299	7500psi Hexane
27	KTHZ60		11/20/07	11/28/07	60	Ambient	72	3.06	52.85	3308	7500psi Hexane
28	KTHZ60		11/20/07	11/28/07	60	Ambient	72	3.09	49.05	3305	7500psi Hexane
29	KTHZ60		11/20/07	11/28/07	60	Ambient	72	3.09	49.43	3325	7500psi Hexane
30	KTHZ60		11/20/07	11/28/07	60	Ambient	72	3.05	51.33	3315	7500psi Hexane
31	KTHZ60		11/20/07	11/28/07	60	Ambient	72	3.12	47.68	3330	7500psi Hexane
32	KTHZ60		11/20/07	11/28/07	60	Ambient	72	2.85	55.51	3335	7500psi Hexane
33	KTHZ60		11/20/07	11/28/07	60	Ambient	72	3.03	49.81	3317	7500psi Hexane

Average		3.02	50.51	3316.60
High		3.12	55.51	3335.00
Low		2.85	47.68	3299.00
SD		0.09	2.27	11.27

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Test No.	BATCH KTHZ60 Formulation	Date Rec	Date Loaded	Date Tested	Ambient Temperature	Conditioned Temperature	Estimated Primer Temperature	Action Time	Pressure	Velocity	Notes
	Fluorinert				°F	°F	°F	ms	ksi	fps	
1	M52A3B1			11/28/07	60	Ambient	72	2.88	45.25	3255	Checkout 1
14	KTHZ60		11/20/07	11/28/07	60	Ambient	72	3.04	50.95	3322	7000psi Hexane
15	KTHZ60		11/20/07	11/28/07	60	Ambient	72	3.02	47.43	3306	7000psi Hexane
16	KTHZ60		11/20/07	11/28/07	60	Ambient	72	2.90	51.71	3328	7000psi Hexane
17	KTHZ60		11/20/07	11/28/07	60	Ambient	72	3.00	50.57	3318	7000psi Hexane
18	KTHZ60		11/20/07	11/28/07	60	Ambient	72	2.98	50.95	3315	7000psi Hexane
19	KTHZ60		11/20/07	11/28/07	60	Ambient	72	2.99	49.33	3321	7000psi Hexane
20	KTHZ60		11/20/07	11/28/07	60	Ambient	72	3.09	49.05	3310	7000psi Hexane
21	KTHZ60		11/20/07	11/28/07	60	Ambient	72	3.06	49.81	3323	7000psi Hexane
22	KTHZ60		11/20/07	11/28/07	60	Ambient	72	3.03	50.57	3315	7000psi Hexane
						Average		3.01	50.04	3317.56	
						High		3.09	51.71	3328.00	
						Low		2.90	47.43	3306.00	
						SD		0.05	1.29	6.84	

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BATCH KTHZ60	Al/MoO$_3$ Molar Ratio	Wt% Al/MoO$_3$	Wt% AN	Wt% Kel-F	Wt% C							Notes
	2.63	66%	30%	2%	2%							
Test No.	Formulation Fluorinert	Date Rec	Date Loaded	Date Tested	Ambient Temperature °F	Conditioned Temperature °F	Estimated Primer Temperature °F	Action Time ms	Pressure ksi	Velocity fps		Notes
1	M52A3B1			11/28/07	60	Ambient	72	2.88	45.25	3255		Checkout 1
4	KTHZ60		11/20/07	11/28/07	60	Ambient	72	3.13	51.71	3316		6500psi Hexane
5	KTHZ60		11/20/07	11/28/07	60	Ambient	72	3.08	49.05	3319		6500psi Hexane
6	KTHZ60		11/20/07	11/28/07	60	Ambient	72	2.98	50.19	3302		6500psi Hexane
7	KTHZ60		11/20/07	11/28/07	60	Ambient	72	3.05	48.67	3308		6500psi Hexane
8	KTHZ60		11/20/07	11/28/07	60	Ambient	72	N/A	N/A	N/A		6500psi Hexane
	KTHZ60		11/20/07	11/28/07	60	Ambient	72	N/A	N/A	N/A		6500psi Hexane
9	KTHZ60		11/20/07	11/28/07	60	Ambient	72	3.10	52.47	3301		6500psi Hexane
10	KTHZ60		11/20/07	11/28/07	60	Ambient	72	2.98	50.19	3303		6500psi Hexane
11	KTHZ60		11/20/07	11/28/07	60	Ambient	72	2.89	53.61	3315		6500psi Hexane
12	KTHZ60		11/20/07	11/28/07	60	Ambient	72	3.06	49.43	3306		6500psi Hexane

Average		3.03	50.67	3308.75
High		3.13	53.61	3319.00
Low		2.89	48.67	3301.00
SD		0.08	1.76	7.01

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KTHZ 60	Al/MoO$_3$ Molar Ratio	Wt% Al/MoO$_3$	Wt% AN	Wt% Kel-F	Wt% C							
	2.63	66%	30%	2%	2%							

Test No.	Formulation Fluorinert	Date Rec	Date Loaded	Date Tested	Ambient Temperature °F	Conditioned Temperature °F	Estimated Primer Temperature °F	Action Time ms	Pressure ksi	Velocity fps	Notes
1	M52A3B1			11/28/07	60	Ambient	72	2.88	45.25	3255	Checkout 1
2	KTHZ60		11/20/07	11/28/07	60	Ambient	72	3.05	50.95	3298	6000psi Dry
3	KTHZ60		11/20/07	11/28/07	60	Ambient	72	3.03	49.81	3289	6500psi Dry
13	KTHZ60		11/20/07	11/28/07	60	Ambient	72	3.01	49.05	3295	7000psi Dry
23	KTHZ60		11/20/07	11/28/07	60	Ambient	72	3.02	50.19	3309	7500psi Dry

Average		3.03	50.00	3297.75
High		3.05	50.95	3309.00
Low		3.01	49.05	3289.00
SD		0.02	0.79	8.38

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BATCH KTHY64	Al/MoO$_3$ Molar Ratio	Wt% Al/MoO$_3$	Wt% AN	Wt% Kel-F	Wt% C						
	2.63	66%	30%	2%	2%						
Test No.	Formulation	Date Rec	Date Loaded	Date Tested	Ambient Temperature °F	Conditioned Temperature °F	Estimated Primer Temperature °F	Action Time ms	Pressure ksi	Velocity fps
1	KTHY64			4/12/07	60	Ambient	70	3.07	47.2	3281
2	KTHY64			4/12/07	60	Ambient	70	3.02	48.0	3332
3	KTHY64			4/12/07	60	Ambient	70	3.09	49.8	3325
4	KTHY64			4/12/07	60	Ambient	70	3.09	47.2	3316
5	KTHY64			4/12/07	60	Ambient	70	3.07	47.5	3287
6	KTHY64			4/12/07	60	Ambient	70	2.91	48.3	3302
7	KTHY64			4/13/07	60	-65	-74	3.09	53.2	3184
8	KTHY64			4/13/07	60	-65	-63	3.27	50.9	3182
9	KTHY64			4/13/07	60	-65	-59	3.16	48.0	3185
10	KTHY64			4/13/07	60	-65	-59	N/A	N/A	N/A
						Average		3.09	48.90	3266.00
						High		3.27	53.23	3332.00
						Low		2.91	47.15	3182.00
						SD		0.10	2.05	63.87
						Ambient	0.07	3.04	47.99	3307.17
						LT	0.09	3.17	50.72	3183.67

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BATCH KTHY62	Al/MoO$_3$ Molar Ratio	Wt% Al/MoO$_3$	Wt% AN	Wt% Kel-F	Wt% C						
	2.63	57%	39%	2%	2%						
Test No.	Formulation	Date Rec	Date Loaded	Date Tested	Ambient Temperature	Conditioned Temperature	Estimated Primer Temperature	Action Time	Pressure	Velocity	
					°F	°F	°F	ms	ksi	fps	
1	KTHY62			4/12/07	60	Ambient	70	3.00	50.5	3329	
2	KTHY62			4/12/07	60	Ambient	70	3.15	49.1	3311	
3	KTHY62			4/12/07	60	Ambient	70	2.98	49.8	3354	
4	KTHY62			4/12/07	60	Ambient	70	3.04	48.4	3326	
5	KTHY62			4/12/07	60	Ambient	70	3.10	48.0	3334	
6	KTHY62			4/13/07	60	-65	-65	3.13	48.7	3187	
7	KTHY62			4/13/07	60	-65	-72	3.27	50.9	3204	
8	KTHY62			4/13/07	60	-65	-61	3.05	52.5	3177	
9	KTHY62			4/13/07	60	-65	-61	3.06	48.8	3207	
10	KTHY62			4/13/07	60	-65	-75	3.10	49.1	3217	
						Average		3.09	49.56	3264.60	
						High		3.27	52.47	3354.00	
						Low		2.98	47.98	3177.00	
						SD		0.08	1.38	71.36	
						Ambient	0.07	3.05	49.14	3330.80	
						LT	0.09	3.12	49.99	3198.40	

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BATCH KTHY50A	Al/MoO$_3$ Molar Ratio	Wt% Al/MoO$_3$	Wt% AN	Wt% Kel-F	Wt% C						
	2.72	76%	20%	2%	2%						
1	Formulation	Date Rec	Date Loaded	Date Tested	Ambient Temperature	Conditioned Temperature	Estimated Primer Temperature	Action Time	Pressure	Velocity
2					°F	°F	°F	ms	ksi	fps
3	KTHY50A			4/12/07	60	Ambient	70	3.05	47.6	3313
4	KTHY50A			4/12/07	60	Ambient	70	3.08	47.9	3318
5	KTHY50A			4/12/07	60	Ambient	70	3.14	48.4	3329
6	KTHY50A			4/12/07	60	Ambient	70	3.07	47.9	3316
7	KTHY50A			4/12/07	60	Ambient	70	3.03	48.0	3315
						Average		3.07	47.95	3318.20
						High		3.14	48.35	3329.00
						Low		3.03	47.62	3313.00
						SD		0.04	0.26	6.30

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STANDARD FORMULATION WITH Al/MoO$_3$ Ratio of 2.51

BATCH KTHY50	Al/MoO$_3$ Molar Ratio	Wt% Al/MoO$_3$	Wt% AN	Wt% Kel-F	Wt% C						
	2.51	66%	30%	2%	2%						
Test No.	Formulation	Date Rec	Date Loaded	Date Tested	Ambient Temperature	Conditioned Temperature	Estimated Primer Temperature	Action Time	Pressure	Velocity
	Fluorinert				^{o}F	^{o}F	^{o}F	ms	ksi	fps
1	M52A3B1		10/25/07	10/31/07	75	Ambient	75	2.74	N/A	3336
2	M52A3B1		10/25/07	10/31/07	75	Ambient	75	2.81	48.29	3323
3	M52A3B1		10/25/07	10/31/07	75	-65	-72	3.01	43.73	3151
4	KTHZ50		10/25/07	10/31/07	75	Ambient	75	3.26	49.05	3336
5	KTHZ50		10/25/07	10/31/07	75	Ambient	75	3.28	50.72	3229
6	KTHZ50		10/25/07	10/31/07	75	Ambient	75	3.61	50.72	3368
7	KTHZ50		10/25/07	10/31/07	75	Ambient	75	3.21	50.51	3260
8	KTHZ50		10/25/07	10/31/07	75	Ambient	75	3.12	48.71	3408
9	KTHZ50		10/25/07	10/31/07	75	-65	-65	3.36	46.18	3146
10	KTHZ50		10/25/07	10/31/07	75	-65	-66	3.31	46.01	3192
11	KTHZ50		10/25/07	10/31/07	75	-65	-67	3.27	47.01	3254
12	KTHZ50		10/25/07	10/31/07	75	-65	-62	3.21	48.29	3146
13	KTHZ50		10/25/07	10/31/07	75	-65	-69	3.29	46.27	3123
						Average		3.29	48.35	3246.20
						High		3.61	50.72	3408.00
						Low		3.12	46.01	3123.00
						SD		0.13	1.91	98.78
						Average	Ambient	3.30	-65	3.29
						SD		0.19		0.05

ALTERNATIVE MIXING PROCESSES AND MICRON METAL POWDER ADDITION

BATCH KTHY84	Al/MoO$_3$ Molar Ratio	Wt% Al/MoO$_3$/Kel-F	Wt% AN		Wt% C					
	2.75	73%	25%		2%					
Test No.	Formulation	Date Rec	Date Loaded	Date Tested	Ambient Temperature °F	Conditioned Temperature °F	Estimated Primer Temperature °F	Action Time ms	Pressure ksi	Velocity fps
1	KTHY84		6/27/07	7/3/07	105	-65	-68	3.58	57.38	3124
2	KTHY84		6/27/07	7/5/07	105	-65	-62	4.15	58.10	3181
3	KTHY84		6/27/07	7/5/07	105	-65	-64	3.21	53.99	3165
4	KTHY84		6/27/07	7/5/07	105	-65	-68	3.40	58.94	3223
5	KTHY84		6/27/07	7/5/07	105	-65	-70	3.38	58.10	3146
6	KTHY84		6/27/07	7/2/07	105	Ambient	75	3.12	58.17	3376
7	KTHY84		6/27/07	7/2/07	105	Ambient	75	3.04	60.99	3329
8	KTHY84		6/27/07	7/2/07	105	Ambient	75	3.08	60.63	3349
9	KTHY84		6/27/07	7/2/07	105	Ambient	75		N/A	N/A
10	KTHY84		6/27/07	7/2/07	105	Ambient	75	3.34	60.27	3241
11	KTHY84		6/27/07	7/3/07	105	Ambient	75	3.27	58.17	3323
12	KTHY84		6/27/07	7/3/07	105	Ambient	75	3.31	58.82	3329

Average		3.35	58.51	3253.27
High		4.15	60.99	3376.00
Low		3.04	53.99	3124.00
SD		0.31	1.91	91.03
Average	Ambient	3.19	-65	3.54
SD		0.13		0.42

Test No.	BATCH KTHY85 Formulation	Al/MoO$_3$ Molar Ratio	Wt% Al/MoO$_3$/Kel-F	Wt% AN		Wt% C	Ti(-325)			
		2.75	63%	25%		2%	10%			

Test No.	Formulation	Date Rec mm/dd/yy	Date Loaded mm/dd/yy	Date Tested mm/dd/yy	Ambient Temperature °F	Conditioned Temperature °F	Estimated Primer Temperature °F	Action Time ms	Pressure ksi	Velocity fps
1	KTHY85		6/28/07	7/5/07	105	-65	-58	3.37	58.94	3151
2	KTHY85		6/28/07	7/5/07	105	-65	-65	3.54	59.18	3208
3	KTHY85		6/28/07	7/5/07	105	-65	-65	3.66	59.70	3157
4	KTHY85		6/28/07	7/5/07	105	-65	-68	3.33	59.54	3181
5	KTHY85		6/28/07	7/5/07	105	-65	-61	3.38	55.86	3151
6	KTHY85		6/28/07	7/2/07	105	Ambient	75	3.16	59.70	3288
7	KTHY85		6/28/07	7/2/07	105	Ambient	75	3.23	59.70	3288
8	KTHY85		6/28/07	7/2/07	105	Ambient	75	3.16	59.32	3327
9	KTHY85		6/28/07	7/2/07	105	Ambient	75	3.05	60.27	3311
10	KTHY85		6/28/07	7/2/07	105	Ambient	75	3.09	59.18	3330
11	KTHY85		6/28/07	7/2/07	105	Ambient	75	3.01	59.90	3320

Average		3.27	59.21	3246.55
High		3.66	60.27	3330.00
Low		3.01	55.86	3151.00
SD		0.21	1.17	76.47
Average	Ambient	3.12	-65	3.46
SD		0.08		0.14

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KTHY86	Al/MoO$_3$ Molar Ratio	Wt% Al/MoO$_3$/Kel-F	Wt% AN		Wt% C	Ti(-325)				
	2.75	68%	25%		2%	5%				
Test No.	Formulation	Date Rec	Date Loaded	Date Tested	Ambient Temperature	Conditioned Temperature	Estimated Primer Temperature	Action Time	Pressure	Velocity
					^{o}F	^{o}F	^{o}F	ms	ksi	fps
1	KTHY86		6/28/07	7/2/07	76	-65	-65	3.51	49.79	3304
2	KTHY86		6/28/07	5/9/07	76	-65	-58	3.94	49.05	3376
3	KTHY86		6/28/07	5/9/07	76	-65	-66	3.89	49.07	3254
4	KTHY86		6/28/07	5/9/07	76	-65	-67	3.32	48.29	3376
5	KTHY86		6/28/07	5/9/07	76	-65	-65	3.27	49.43	3379
6	KTHY86		6/28/07	5/9/07	76	Ambient	75	3.66	48.67	3356
7	KTHY86		6/28/07	5/9/07	76	Ambient	75	3.13	48.35	3320
8	KTHY86		6/28/07	5/9/07	76	Ambient	75	3.37	48.71	3330
9	KTHY86		6/28/07	5/9/07	76	Ambient	75	3.65	47.62	3339
10	KTHY86		6/28/07	5/9/07	76	Ambient	75	3.31	46.54	3330
11	KTHY86		6/28/07	5/9/07	76	Ambient	75	3.12	48.29	3177
12	KTHY86		6/28/07	5/9/07	76	Ambient	75	3.19	47.91	3222

Average		3.45	48.48	3313.58
High		3.94	49.79	3379.00
Low		3.12	46.54	3177.00
SD		0.28	0.87	64.51
Average	Ambient	3.35	-65	3.59
SD		0.23		0.31

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KTHY87	Al/MoO$_3$ Molar Ratio	Wt% Al/MoO$_3$/Kel-F	Wt% AN		Wt% C					
	2.75	88%	10%		2%					
Test No.	Formulation	Date Rec	Date Loaded	Date Tested	Ambient Temperature	Conditioned Temperature	Estimated Primer Temperature	Action Time	Pressure	Velocity
					^{o}F	^{o}F	^{o}F	ms	ksi	fps
1	KTHY87		6/27/07	7/3/07	105	-65	-65	14.41	60.74	3260
2	KTHY87		6/27/07	7/3/07	105	-65	-64	4.73	56.65	3088
3	KTHY87		6/27/07	7/3/07	105	-65	-58	4.21	56.65	3158
4	KTHY87		6/27/07	7/3/07	105	-65	-56	4.80	55.93	3123
5	KTHY87		6/27/07	7/3/07	105	-65	-58	9.27	56.29	3147
6	KTHY87		6/27/07	7/2/07	105	Ambient	75	3.94	59.54	3320
7	KTHY87		6/27/07	7/2/07	105	Ambient	75	3.81	58.10	3272
8	KTHY87		6/27/07	7/2/07	105	Ambient	75	3.66	58.82	3352
9	KTHY87		6/27/07	7/2/07	105	Ambient	75	3.73	59.18	3352
10	KTHY87		6/27/07	7/2/07	105	Ambient	75	3.94	60.27	3329
11	KTHY87		6/27/07	7/2/07	105	Ambient	75	3.38	59.18	3329
12	KTHY87		6/27/07	7/2/07	105	Ambient	75	3.43	60.27	3339
						Average		5.28	58.47	3255.75
						High		14.41	60.74	3352.00
						Low		3.38	55.93	3088.00
						SD		3.29	1.70	98.97
						Average	Ambient	3.70	-65	7.48
						SD		0.23		4.38

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KTHY87	Al/MoO$_3$ Molar Ratio	Wt% Al/MoO$_3$/Kel-F	Wt% AN		Wt% C					
	2.65	68%	30%		2%					
Test No.	Formulation Fluorinert	Date Rec	Date Loaded	Date Tested	Ambient Temperature °F	Conditioned Temperature °F	Estimated Primer Temperature °F	Action Time ms	Pressure ksi	Velocity fps
1	M52A3B1			12/19/07	45	Ambient	72	2.82	42.93	3290
6	KTHZ64		12/3/07	12/19/07	45	Ambient	72	3.42	47.91	3222
7	KTHZ64		12/3/07	12/19/07	45	Ambient	72	3.66	48.35	3292
8	KTHZ64		12/3/07	12/19/07	45	Ambient	72	3.45	48.71	3293
9	KTHZ64		12/3/07	12/19/07	45	Ambient	72	3.38	50.51	3277
10	KTHZ64		12/3/07	12/19/07	45	Ambient	72	3.44	52.09	3292
11	KTHZ64		12/3/07	12/19/07	45	Ambient	72	3.46	51.24	3293
12	KTHZ64		12/3/07	12/19/07	45	Ambient	72	3.46	49.07	3300
13	KTHZ64		12/3/07	12/19/07	45	Ambient	72	3.33	51.33	3287
14	KTHZ64		12/3/07	12/19/07	45	Ambient	72	3.46	50.19	3300
15	KTHZ64		12/3/07	12/19/07	45	Ambient	72	3.40	47.98	3292
16	KTHZ64		12/3/07	12/19/07	45	Ambient	72	3.47	49.79	3317
17	KTHZ64		12/3/07	12/19/07	45	Ambient	72	3.33	52.68	3313
18	KTHZ64		12/3/07	12/19/07	45	Ambient	72	3.44	50.87	3294
19	KTHZ64		12/3/07	12/19/07	45	Ambient	72	3.56	50.87	3283
20	KTHZ64		12/3/07	12/19/07	45	Ambient	72	3.33	48.67	3293
						Average		3.44	50.02	3289.87
						High		3.66	52.68	3317.00
						Low		3.33	47.91	3222.00
						SD		0.09	1.52	21.32

KTHAD13	Al/MoO$_3$ Molar Ratio	Wt% Al/MoO$_3$/Bi$_2$O$_3$	Wt% AN	Wt% Kel-F	Wt% C						
		66%	30%	2%	2%						

Date: 07 May 2009					Test Engineer: D. Herigstad				L. Wheeler	
Test: Green Primer Trials					Firing Officer: C. Lancaster				Gunner: A. Camacho	

Event No.	Time	Peak Pressure (kpsi)	Velocity (fps)	Action Time (ms)	Prop. Wt. (grs)	Proj. Wt. (grs)	Primer Type	Barrel Temp (deg F)	Notes	
16	13:44	52.47	3250	3.00		N/A	M52A3B1	-71.6	Checkout 1	
17	13:48	49.43	3185	4.50	580.0	N/A	KTHAD13	-73.6	Round 15	
18	13:52	47.98	3155	4.19	580.0	N/A	KTHAD13	-62.8	Round 16	
19	13:59	50.82	3253	4.41	580.0	N/A	KTHAD13	-62.6	Round 17	
20	14:02	51.24	3256	34.05	580.0	N/A	KTHAD13	-67.8	Round 18	
21	14:05	47.53	3205	4.17	580.0	N/A	KTHAD13	-62.6	Round 19	
22	14:12	48.35	3136	4.63	580.0	N/A	KTHAD13	-65.8	Round 20	
23	14:15	47.15	3196	4.05	580.0	N/A	KTHAD13	-71.2	Round 21	
24	14:27	49.43	3181	4.60	580.0	N/A	KTHAD13	-67.8	Round 22	
25	14:30	55.89	3293	12.22	580.0	N/A	KTHAD13	-70.6	Round 23	
26	14:33	40.86	3189	4.53	580.0	N/A	KTHAD13	-64.8	Round 24	
27	14:39	45.70	3221	5.07	580.0	N/A	KTHAD13	-62.4	Round 25	
28	14:14	44.43	3209	4.82	580.0	N/A	KTHAD13	-62.2	Round 26	
29	14:48	47.62	3210	4.13	580.0	N/A	KTHAD13	-61.4	Round 27	
30	14:51	50.15	3310	2.85		N/A	M52A3B1	-61.2	Checkout 2	
				7.34						
			STDDEV	8.08						

AMMUNITION LOT NUMBER: KTHAD13 Hexane   OAT 92

Propellant: WC 868

Projectiles: PGU-27A/B

Loaded By: A. Camacho / L. Wheeler

Ammunition on this sheet conditioned to -65 deg. F

Barrel Serial Number: 004

Breech Block Serial Number: 008

EFFECT OF ADDITION OF Al(H30)

KTHY66	Al/MoO$_3$ Molar Ratio	Wt% Al/MoO$_3$	Wt% AN	Wt% Kel-F	Wt% C	Al(H30)					
	2.63	62%	24%	2%	2%	10%					
Test No.	Formulation	Date Rec	Date Loaded	Date Tested	Ambient Temperature	Conditioned Temperature	Estimated Primer Temperature		Action Time	Pressure	Velocity
					°F	°F	°F		ms	ksi	fps
1	KTHY66			5/9/07	76	Ambient	75		3.08	49.1	3355
2	KTHY66			5/9/07	76	Ambient	75		3.26	49.8	3436
3	KTHY66			5/9/07	76	Ambient	75		3.15	49.4	3445
4	KTHY66			5/9/07	76	Ambient	75		3.51	50.5	3330
5	KTHY66			5/9/07	76	Ambient	75		3.21	48.7	3330
6	KTHY66			5/9/07	76	Ambient	75		3.25	49.4	3362
7	KTHY66			5/9/07	76	Ambient	75		3.07	49.7	3363
8	KTHY66			5/9/07	76	-65	-73		3.06	49.1	3208
9	KTHY66			5/9/07	76	-65	-80		3.45	47.6	3247
10	KTHY66			5/9/07	76	-65	-69		3.24	48.3	3222
11	KTHY66			5/9/07	76	-65	-62		3.25	47.6	3208
12	KTHY66			5/9/07	76	-65	-60		3.71	44.7	3223
						Average			3.27	48.66	3310.75
						High			3.71	50.51	3445.00
						Low			3.06	44.70	3208.00
						SD			0.20	1.52	86.61
						Average	Ambient		3.22	-65	3.34
						SD			0.15		0.25

The role of large particles in the LFEP was investigated by adding Al(H30). The addition of 10 wt% resulted in a longer average AUR-AT and larger standard deviation.

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KTHY73	Al/MoO$_3$ Molar Ratio	Wt% Al/MoO$_3$	Wt% AN	Wt% Kel-F	Wt% C	Al(H30)			
	2.73	51%	25%	2%	2%	20%			
Test No.	Formulation	Date Rec	Date Loaded	Date Tested	Ambient Temperature	Conditioned Temperature	Estimated Primer Temperature	Action Time	Pressure	Velocity
					°F	°F	°F	ms	ksi	fps
1	KTHY73			5/9/07	76	Ambient	75	3.51	49.8	3304
2	KTHY73			5/9/07	76	Ambient	75	3.94	49.1	3376
3	KTHY73			5/9/07	76	Ambient	75	3.89	49.1	3254
4	KTHY73			5/9/07	76	Ambient	75	3.32	48.3	3376
5	KTHY73			5/9/07	76	Ambient	75	3.27	49.4	3379
6	KTHY73			5/9/07	76	Ambient	75	3.66	48.7	3356
7	KTHY73			5/9/07	76	Ambient	75	3.13	48.4	3320
8	KTHY73			5/9/07	76	Ambient	75	3.37	48.7	3330
9	KTHY73			5/9/07	76	Ambient	75	3.65	47.6	3339
10	KTHY73			5/9/07	76	Ambient	75	3.31	46.5	3330
11	KTHY73			5/9/07	76	-65	-61	3.12	48.3	3177
12	KTHY73			5/9/07	76	-65	-64	3.19	47.91	3222
13	KTHY73			5/9/07	76	-65	-65	3.40	47.26	3229
14	KTHY73			5/9/07	76	-65	-64	3.23	47.53	3216
15	KTHY73			5/9/07	76	-65	-66	3.29	48.35	3123
16	KTHY73			5/9/07	76	-65	-64	3.22	49.43	3187
17	KTHY73			5/9/07	76	-65	-65	3.40	45.82	3181
18	KTHY73			5/9/07	76	-65	-67	3.46	44.87	3148
19	KTHY73			5/9/07	76	-65	-62	3.22	42.59	3151
20	KTHY73			5/9/07	76	-65	-62	3.49	46.01	3093
						Average		3.40	47.68	3254.55
						High		3.94	49.79	3379.00
						Low		3.12	42.59	3093.00
						SD		0.23	1.77	93.24
						Average	Ambient	3.51	-65	3.30
						SD		0.27		0.13

EFFECT OF TEFLON ON AUR-AT

KTHZ20	Al/MoO$_3$ Molar Ratio	Wt% Al/MoO$_3$	Wt% AN	Wt% Kel-F	Wt% C	Wt% Teflon					
	2.63	64%	29%	2%	2%	3%					
Test No.	Formulation	Date Rec	Date Loaded	Date Tested	Ambient Temperature	Conditioned Temperature	Estimated Primer Temperature	Action Time	Pressure	Velocity
					^{o}F	^{o}F	^{o}F	ms	ksi	fps
1	M52A3B1		8/29/07	9/6/07	85	Ambient	75	2.82	47.91	3350
2	KTHZ20		8/29/07	9/6/07	85	Ambient	75	N/A	49.43	3326
3	KTHZ20		8/29/07	9/6/07	85	Ambient	75	26.00	52.09	3338
4	KTHZ20		8/29/07	9/6/07	85	Ambient	75	3.82	48.67	3259
5	KTHZ20		8/29/07	9/6/07	85	Ambient	75	4.53	50.57	3241
6	KTHZ20		8/29/07	9/6/07	85	Ambient	75	4.02	49.81	3324
7	KTHZ20		8/29/07	9/6/07	85	Ambient	75	5.77	51.33	3336
8	KTHZ20		8/29/07	9/6/07	85	Ambient	75	4.00	50.19	3376
9	KTHZ20		8/29/07	9/6/07	85	Ambient	75	4.26	50.15	3335
10	KTHZ20		8/29/07	9/6/07	85	Ambient	75	57.90	52.09	3326
11	KTHZ20		8/29/07	9/6/07	85	Ambient	75	4.70	50.19	3345
						Average		12.78	50.45	3320.60
						High		57.90	52.09	3376.00
						Low		3.82	48.67	3241.00
						SD		18.37	1.11	40.29

The addition of 3wt% Teflon dramatically increases the average AUR-AT to unacceptable levels. The high stability of the C-F bonds causes an ignition delay.

ALEX/MoO₃ BASED LFEP

KTHAB26 ALEX	Al/MoO₃ Molar Ratio	Wt% Al/MoO₃	Wt% AN	Wt% Kel-F	Wt% C					
	3.02	66%	30%	2%	2%					
Date: 07 October 2008					Test Engineer: E. Gogley				Instrumentation: L. Wheeler	
Test: Green Primer Trials					Firing Officer: C. Lancaster				Gunner: S. Ford / A. Camacho IT	
Event No.	Time	Peak Pressure (kpsi)	Velocity (fps)	Action Time (ms)	Prop. Wt. (grs)	Proj. Wt. (grs)	Primer Type	Barrel Temp (deg F)	Notes	
1	1034	46.54	3359	2.8			M52A3B1	Amb	Checkout 1	
2	1044	56.29	3272	3.77	580.01		KTHAB26	Amb	Round 18	
3	1050	-	-	-	580.06		KTHAB26	Amb	Round 19, Instrumentation Error	
4	1053	-	-	-	580.02		KTHAB26	Amb	Round 11, Instrumentation Error	
5	1100	49.43	3306	3.31	580.03		KTHAB26	Amb	Round 15	
6	1105	50.15	3347	3.29	580.05		KTHAB26	Amb	Round 22	
7	1109	48.71	3338	3.34	580.03		KTHAB26	Amb	Round 8	
8	1112	51.71	3339	3.64	580.01		KTHAB26	Amb	Round 27	
9	1116	50.15	3342	3.29	580.08		KTHAB26	Amb	Round 9	
10	1118	51.60	3344	3.51	580.02		KTHAB26	Amb	Round 17	
									3.31	
									3.29	
									3.34	
			STDDEV	0.02					3.64	

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			STDDEV	0.13				3.29	
								3.51	
								3.42	
			STDDEV	0.19				3.55	
AMMUNITION LOT NUMBER: KTHAB26 Hexane						OAT 71F		3.68	
Propellant: WC 868								3.44	
Projectiles: PGU-27A/B								3.84	
Loaded By: A. Camacho L. Wheeler									
								3.48	
Barrel Serial Number: 004								0.18	
Breech Block Serial Number: 008									
			Ambient	Avg	3.45	LT	4.62		
				SD	0.19		1.23		

KTHAB26 ALEX	Al/MoO$_3$ Molar Ratio	Wt% Al/MoO$_3$	Wt% AN	Wt% Kel-F	Wt% C				
	3.02	66%	30%	2%	2%				

B52 BALLISTIC TEST LABORATORY
Code 478200D

Date: 08 October 2008					Test Engineer: E. Gogley				Instrumentation: L. Wheeler
Test: Green Primer Trials					Firing Officer: C. Lancaster				Gunner: S. Ford / A. Camacho IT

Event No.	Time	Peak Pressure (kpsi)	Velocity (fps)	Action Time (ms)	Prop. Wt. (grs)	Proj. Wt. (grs)	Primer Type	Barrel Temp (deg F)	Notes
1	1319	57.03	3301	3.04			M52A3B1	-76	Checkout round
2	1324	49.79	3131	3.82	580.04		KTHAB26	-81	Round 14
3	1328	51.33	3165	4.75	580.03		KTHAB26	-71	Round 04
4	1332	50.57	3185	3.93	580.05		KTHAB26	-54	Round 13
5	1341	50.72	3201	5.14	580.05		KTHAB26	-67	Round 01
6	1345	48.29	3147	4.57	580.04		KTHAB26	-82	Round 30
7	1348	51.60	3116	4.45	580.05		KTHAB26	-73	Round 03
8	1352	50.57	3164	8.58	580.03		KTHAB26	-66	Round 23
9	1358				580.09		KTHAB26	-65	Round 29, Misfire
10	1412	51.33	3130	4.99	580.07		KTHAB26	-69	Round 07
11	1415	49.07	3136	4.39	580.02		KTHAB26	-65	Round 06
12	1419	52.47	3173	4.15	580.01		KTHAB26	-61	Round 05
13	1426	53.04	3192	3.99	580.07		KTHAB26	-64	Round 24
14	1429	52.85	3178	3.88	580.02		KTHAB26	-73	Round 10
15	1432	51.33	3151	4.59	580.06		KTHAB26	-65	Round 20
16	1435	57.02	3312	3.50	580.09		KTHAB26	-44	Round 29
		LT	Average	4.62					
			STDDEV	1.23					

AMMUNITION LOT NUMBER: KTHAB26 Hexane   OAT 89F

Propellant: WC 868

Projectiles: PGU-27A/B

Loaded By: A. Camacho L. Wheeler

All rounds this page conditioned to -53.9 deg C

Barrel Serial Number: 004

Breech Block Serial Number: 008

KTHAD60D - Effect of 3 Weight Percent Carbon on the AUR-AT (Slightly Higher AUR-ATs)

Date: 25 November 2009				Test Engineer: D. Herigstad						Instrumentation: L. Wheeler
Test: Green Primer Trials				Firing Officer: C. Lancaster						Gunner: A. Camacho
Event No.	Time	Peak Pressure (kpsi)	Velocity (fps)	Action Time (ms)	Prop. Wt. (grs)	Proj. Wt. (grs)	Primer Type	Barrel Temp (deg F)	Resistance in Ohms	Notes
1	8:17	41.83	3354	2.50		N/A	M52A3B1	Amb		Check out Round 1 Lot # OL01F192-002 PGU-27 A/B
2	8:38	47.98	3388	3.09	580.00	N/A	KTHAD60B	Amb	5.898	Round 1
3	8:41	47.91	3388	3.13	580.00	N/A	KTHAD60B	Amb	7.282	Round 2
4	8:43	47.62	3382	3.10	580.00	N/A	KTHAD60B	Amb	6.676	Round 3
5	8:45	47.98	3385	3.12	580.00	N/A	KTHAD60B	Amb	6.232	Round 4
6	8:48	48.35	3384	3.35	580.00	N/A	KTHAD60B	Amb	6.841	Round 5
7	8:50	49.07	3407	3.23	580.00	N/A	KTHAD60B	Amb	5.350	Round 6
8	8:52	47.26	3382	3.22	580.00	N/A	KTHAD60B	Amb	6.981	Round 7
9	8:54	47.26	3382	3.15	580.00	N/A	KTHAD60B	Amb	6.208	Round 8
10	8:56	46.62	3380	3.22	580.00	N/A	KTHAD60B	Amb	7.340	Round 9
11	8:58	47.91	3409	3.09	580.00	N/A	KTHAD60B	Amb	6.974	Round 10
12	9:00	47.98	3409	3.12	580.00	N/A	KTHAD60B	Amb	76.300	Round 11
13	9:03	48.33	3387	3.44	580.00	N/A	KTHAD60B	Amb	65.560	Round 12
14	9:05	48.35	3381	3.30	580.00	N/A	KTHAD60B	Amb	57.560	Round 13
15	9:07	47.62	3400	3.33	580.00	N/A	KTHAD60B	Amb	53.860	Round 14
16	9:09	47.98	3385	3.28	580.00	N/A	KTHAD60B	Amb	62.290	Round 15
AVE		47.88	3389.9	3.21						
			S D.	0.10						
AMMUNITION LOT NUMBER: KTHAD60B Ammo temp 73F OAT 51F										Old Die
Propellant: WC 868										No Bake
Projectiles: PGU-27/B										Dry Load
Loaded By: A. Camacho, L. Wheeler										Breech Block Serial Number: 008
										Barrel Serial Number: 004

200

KTHAB66 – Alternative Mixing Process, and ALEX in Place of Al(80nm) (Much Longer AUR-ATs)

Date: 28 October 2008					Test Engineer: E. Gogley			Instrumentation: L. Wheeler	
Test: Green Primer Trials					Firing Officer: C. Lancaster			Gunner: S. Ford / A. Camacho (IT)	

Event No.	Time	Peak Pressure (kpsi)	Velocity (fps)	Action Time (ms)	Prop. Wt. (grs)	Proj. Wt. (grs)	Primer Type	Barrel Temp (deg F)	Notes
1	14:24		3356	N/A			M52A3B1	Ambient	Checkout 1
2	14:38	48.29	3403	2.87			M52A3B1	Ambient	Checkout 2
3	14:46	56.69	3333	3.65	580.01		KTHAB66	Ambient	Round 1
4	14:49	53.23	3372	3.25	580.04		KTHAB66	Ambient	Round 2
5	14:52	55.29	3349	4.39	580.02		KTHAB66	Ambient	Round 3
6	14:55	56.37	3327	3.61	580.03		KTHAB66	Ambient	Round 4
7	14:57	54.49	3347	4.05	580.04		KTHAB66	Ambient	Round 5
8	15:00	55.13	3325	3.29	580.04		KTHAB66	Ambient	Round 6
9	15:02	55.12	3362	4.75	580.05		KTHAB66	Ambient	Round 7
10	15:04	56.88	3342	3.62	580.03		KTHAB66	Ambient	Round 8
11	15:07	54.14	3326	3.40	580.02		KTHAB66	Ambient	Round 9
12	15:09	53.69	3336	3.41	580.02		KTHAB66	Ambient	Round 10
13	15:11	55.51	3355	3.42	580.01		KTHAB66	Ambient	Round 11
14	15:13	58.10	3369	3.41	580.04		KTHAB66	Ambient	Round 12
15	15:16	55.17	3356	3.81	580.04		KTHAB66	Ambient	Round 13
16	15:18	54.09	3365	3.29	580.00		KTHAB66	Ambient	Round 14
17	15:20	55.51	3397	3.71	580.01		KTHAB66	Ambient	Round 15
				3.67	4.75				
			STDDEV	0.43	3.25				

AMMUNITION LOT NUMBER: KTHAB66 Hexane
Propellant: WC 868
Projectiles: PGU-27A/B
Ammo brought to you by: A. Camacho L. Wheeler
Barrel Serial Number: 004
Breech Block Serial Number: 008

				Ambient	Average	3.67	LT	4.88	
					SD	0.43		2.26	

Test: Green Primer Trials					Firing Officer: C. Lancaster				Gunner: S. Ford / A. Camacho (IT)
Event No.	Time	Peak Pressure (kpsi)	Velocity (fps)	Action Time (ms)	Prop. Wt. (grs)	Proj. Wt. (grs)	Primer Type	Barrel Temp (deg F)	Notes
1	1237						M52A3B1	-65	Checkout 1, No fire
2	1251	48.67	3343	2.88			M52A3B1	-42	Ambient temperature round
3	1303	54.83	3187	4.23	580.01		KTHAB66	-70	Round 16
4	1308	52.81	3181	4.45	580.03		KTHAB66	-77	Round 17
5	1311	51.33	3193	4.06	580.03		KTHAB66	-71	Round 18
6	1314	51.96	3178	4.42	580.00		KTHAB66	-61	Round 19
7	1321	53.80	3149	4.06	580.01		KTHAB66	-69	Round 20
8	1324	53.78	3166	12.80	580.02		KTHAB66	-73	Round 21
9	1328	53.46	3152	4.08	580.00		KTHAB66	-66	Round 22
10	1330	51.33	3159	4.03	580.04		KTHAB66	-58	Round 23
11	1337	52.38	3180	4.24	580.00		KTHAB66	-64	Round 24
12	1340	50.57	3178	3.8	580.03		KTHAB66	-70	Round 25
13	1343	52.09	3176	5.37	580.04		KTHAB66	-60	Round 26
14	1350	50.55	3161	5.87	580.01		KTHAB66	-64	Round 27
15	1352	51.2	3135	4.18	580.01		KTHAB66	-70	Round 28
16	1355	52.08	3165	3.85	580.02		KTHAB66	-65	Round 29
17	1357	53.23	3189	3.82	580.03		KTHAB66	-59	Round 30
			AVE	4.88					
				2.26					
			STDDEV	2.26					

AMMUNITION LOT NUMBER: KTHAB66 Hexane

Propellant: WC 868

Projectiles: PGU-27A/B

Ammo brought to you by: A. Camacho L. Wheeler

Ammo and MANN BBL conditioned to -65 deg

Barrel Serial Number: 004

Breech Block Serial Number: 008

KTHAB67 – ALEX/MoO$_3$ Led to Higher AUR-ATs and Larger Scatter

KTHAB67	Al/MoO$_3$ Molar Ratio	Wt% Al/MoO$_3$	Wt% AN	Wt% Kel-F	Wt% C				
	2.89	65%	31%	2%	2%				

| Date: 13 January 2009 | | | | | Test Engineer: S. Ford | | | Instrumentation: S. Ford | |
| Test: Green Primer Trials | | | | | Firing Officer: C. Lancaster | | | Gunner: A. Camacho, L. Wheeler (IT) | |

Event No.	Time	Peak Pressure (kpsi)	Velocity (fps)	Action Time (ms)	Prop. Wt. (grs)	Proj. Wt. (grs)	Primer Type	Barrel Temp (deg F)	Notes
1	13:58	50.38	3312	2.81	N/A	N/A	M55	Amb	M55 Series
2	14:00	50.19	3347	2.69	N/A	N/A	M52A3B1	Amb	Checkout 1
3	14:02	55.57	3330	3.18	580.00	N/A	KTHAB67	Amb	Round 16
4	14:05	53.80	3309	3.23	580.04	N/A	KTHAB67	Amb	Round 17
5	14:07	55.17	3375	3.04	580.00	N/A	KTHAB67	Amb	Round 18
6	14:10	53.04	3336	3.80	580.02	N/A	KTHAB67	Amb	Round 19
7	14:14	55.21	3329	3.98	580.02	N/A	KTHAB67	Amb	Round 20
8	14:17	52.17	3305	3.92	580.01	N/A	KTHAB67	Amb	Round 21
9	14:20	53.23	3348	3.91	580.00	N/A	KTHAB67	Amb	Round 22
10	14:24	54.43	3349	4.36	580.04	N/A	KTHAB67	Amb	Round 23
11	14:27	51.20	3301	3.62	580.02	N/A	KTHAB67	Amb	Round 24
12	14:32	51.60	3345	3.80	580.04	N/A	KTHAB67	Amb	Round 25
13	14:35	53.14	3337	3.51	580.01	N/A	KTHAB67	Amb	Round 26
14	14:38	51.06	3492	3.41	580.02	N/A	KTHAB67	Amb	Round 27
15	14:41	51.06	3355	4.21	580.00	N/A	KTHAB67	Amb	Round 28
16	14:45	51.2	3455	3.64	580.03	N/A	KTHAB67	Amb	Round 29
17	14:47	51.2	3329	3.29	580.01	N/A	KTHAB67	Amb	Round 30
18	14:50	53.99	3370	3.35	580.03	N/A	KTHAB67	Amb	Round 31
			Avg AT	3.64					
			SD	0.38					

AMMUNITION LOT NUMBER: KTHAB67 Hexane   OAT 66F

Propellant: WC 868

Projectiles: PGU-27A/B

203

Loaded By: A. Camacho, L. Wheeler

Barrel Serial Number: 004
Breech Block Serial Number: 008

KTHAF80 – Effects of LFEP Aging for 1 Year as A Dry Solid – AUR-AT Increased to 3.98 ms

KTHAF80	M2671	Wt% Al/MoO$_3$	Wt% AN	Wt% Kel-F	Wt% C					LFEP Aged for 1 Year Before Pressing
	2.8	74%	22%	2%	2%					

										Instrumentation: L. Wheeler
Date: 2 February 2011						Test Engineer: A. Farmer				
Test: Green Primer Trials						Firing Officer: C. Lancaster				Gunner: A. Camacho
Event No.	Round No.	Time	Peak Pressure (kpsi)	Velocity (fps)	Action Time (ms)	Prop. Wt. (grs)	Proj. Wt. (grs)	Primer Type	Barrel Temp (deg F)	Notes
1	CO1	13:29	39.92	3382	0	580.00		M52A3B1		INSTRUMENTATION ERROR
2	CO2	13:39	36.50	3410	2.7	580.00		M52A3B1		
3	1	13:43	46.01	3433	0.00	580.00		KTHAF80		INSTRUMENTATION ERROR
4	CO3	13:59	39.92	3396	0.00	580.00		M52A3B1		INSTRUMENTATION ERROR
5	CO4	14:11	38.59	3399	0.00	580.00		M52A3B1		INSTRUMENTATION ERROR
6	CO5	14:22	38.40	3363	2.98	580.00		M52A3B1		
7	CO6	14:27	36.79	3391	2.84	580		M52A3B1		
8	2	14:29	49.81	3464	0.00	580.00		KTHAF80		INSTRUMENTATION ERROR
9	CO7	14:33	37.87	3384	2.88	580.00		M52A3B1		
10	3	14:37	45.46	3415	3.84	580.00		KTHAF80		
11	4	14:41	47.15	3436	5.12	580.00		KTHAF80		
12	5	14:44	47.53	3431	3.54	580.00		KTHAF80		
13	6	15:15	48.29	3445	3.69	580.00		KTHAF80		
14	7	15:17	48.35	3445	3.85	580.00		KTHAF80		
15	8	15:20	45.1	3405	3.95	580.00		KTHAF80		
16	9	15:22	46.9	3426	4.13	580.00		KTHAF80		
17	10	15:24	47.15	3425	4.26	580.00		KTHAF80		

18	11	15:26	47.62	3441	3.83	580.00		KTHAF80		
19	12	15:29	47.62	3436	4.03	580.00		KTHAF80		
20	13	15:32	49.07	3437	4.05	580.00		KTHAF80		
21	14	15:36	47.62	3438	4.13	580.00		KTHAF80		
22	15	15:38	46.54	3436	3.37	580		KTHAF80		
	(6-15)		Ave.	3433	3.98					
	(6-15)		SD	12	0.42					

AMMUNITION LOT NUMBER: KTHAF80   OAT 47 Deg F

Propellant: WC 868

Projectiles: PGU-27/B

Loaded By: A. Camacho, L. Wheeler

Barrel Serial Number: 004

Breech Block Serial Number: 008

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NEW VERSUS OLD DIE SET

KTHAD31B	Al/MoO$_3$ Molar Ratio	Wt% Al/MoO$_3$	Wt% AN	Wt% Kel-F	Wt% C				
	2.69	57%	39%	2%	2%				

Test: Green Primer Trials					Firing Officer: C. Lancaster			Gunner: A. Camacho	
Event No.	Time	Peak Pressure (kpsi)	Velocity (fps)	Action Time (ms)	Prop. Wt. (grs)	Proj. Wt. (grs)	Primer Type	Barrel Temp (deg F)	Notes
13	12:05	42.17	3326				M52A3B1		Checkout 1 Inst. Glitch
14	12:19	41.83	3358	2.7			M52A3B1		Checkout 2
15	12:25	53.61	3321	24.7			KTHAD31B		Round 11
16	12:28	52.09	3333	32.8			KTHAD31B		Round 12
17	12:31	53.61	3328	40.86			KTHAD31B		Round 13
18	12:33	52.47	3330	35.57			KTHAD31B		Round 14
19	12:36	53.46	3333	13.99			KTHAD31B		Round 15
20	12:38	50.95	3336	46.17			KTHAD31B		Round 16
21	12:41	52.32	3317	32.54			KTHAD31B		Round 17
22	12:45	53.61	3343	31.04			KTHAD31B		Round 18
23	12:47	53.23	3389	21.12			KTHAD31B		Round 19
24	12:50	53.76	3361	41.42			KTHAD31B		Round 20
				32.021					
			STDDEV	9.88					
AMMUNITION LOT NUMBER: KTHAD31B Hexane					OAT 99 F				
Propellant: WC 868									
Projectiles: PGU-27A/B									
Loaded By: A. Camacho / L. Wheeler									
Barrel Serial Number: 004									
Breech Block Serial Number: 008									

B52 BALLISTIC TEST LABORATORY
Code 478200D

KTHAD31B	Al/MoO$_3$ Molar Ratio	Wt% Al/MoO$_3$	Wt% AN	Wt% Kel-F	Wt% C			NEW DIE SET	
	2.63	55%	39%	3%	3%				
Date: 02 July 09						Test Engineer: D. Herigstad		Instrumentation: L. Wheeler	
Test: Green Primer Trials						Firing Officer: C. Lancaster		Gunner: A. Camacho	

Event No.	Time	Peak Pressure (kpsi)	Velocity (fps)	Action Time (ms)	Prop. Wt. (grs)	Proj. Wt. (grs)	Primer Type	Barrel Temp (deg F)	Notes	
1	10:11	46.18	3407	2.7			M52A3B1	Amb	Checkout 1	
2	10:15		3360	5.3	580.0		KTHAD31B		Round 1 Inst. Glitch	
3	10:37	52.49	3351	6.60	580.0		KTHAD31B		Round 2	
4	12:25	46.18	3334	5.10	580.0		KTHAD31B		Round 3	
5	12:29		3343		580.0		KTHAD31B		Round 4 Inst. Glitch	
6	12:42	53.99	3334	5.13	580.0		KTHAD31B		Round 5	
7	12:47	51.60	3318	36.28	580.0		KTHAD31B		Round 6	
8	12:55	51.41	3340	21.17	580.0		KTHAD31B		Round 7	
9	12:58	50.19	3342	19.07	580.0		KTHAD31B		Round 8	
10	13:00				580.0		KTHAD31B		Round 9 NO FIRE 2 ATTEMPTS	
11	13:10	46.01	3388	2.7			M52A3B1		Checkout 2	
12	13:16	54.75	3383	21.9	580.0		KTHAD31B		Round 10	
			STDDEV	11.98						
AMMUNITION LOT NUMBER: KTHAD31B Hexane						OAT 90 F				
Propellant: WC 868										
Projectiles: PGU-27A/B										
Loaded By: A. Camacho / L. Wheeler										
Barrel Serial Number: 004										
Breech Block Serial Number: 008										

B52 BALLISTIC TEST LABORATORY
Code 478200D

KTHAD31B	Al/MoO$_3$ Molar Ratio	Wt% Al/MoO$_3$	Wt% AN	Wt% Kel-F	Wt% C				OLD DIE SET
	2.63	55%	39%	3%	3%				

									Instrumentation: L. Wheeler
Date: 20 June 2011				Test Engineer: D. Herigstad					
Test: Green Primer Trials				Firing Officer: C. Lancaster					Gunner: A. Camacho

Event No.	Time	Peak Pressure (kpsi)	Velocity (fps)	Action Time (ms)	Prop. Wt. (grs)	Proj. Wt. (grs)	Primer Type	Barrel Temp (deg F)	Round Number	Notes
1	11:17	43.88	3647	3.21			KTHAD31B		1D	DRY LOADING, No BAKE
2	11:21	50.72	3451	3.52			KTHAD31B		2D	
3	11:25	46.77	3467	3.40			KTHAD31B		3D	
4	11:27	42.21	3462	3.25			KTHAD31B		4D	
5	11:30	43.54	3476	3.25			KTHAD31B		5D	
6	15:10	45.25	3494	3.19			KTHAD31B		6D	
7	15:13	46.77	3486	3.17			KTHAD31B		7D	
8	15:15	43.88	3494	3.27			KTHAD31B		8D	
9	15:18	44.56	3498	3.69			KTHAD31B		9D	
10	15:20	46.54	3502	3.14			KTHAD31B		10D	
11	15:23	45.25	3506	3.16			KTHAD31B		11D	
12	15:25	45.82	3509	3.33			KTHAD31B		12D	
13	15:28	42.17	3504	3.18			KTHAD31B		13D	
14	15:30	48.35	3526	3.24			KTHAD31B		14D	
15	15:32	45.63	3493	3.30			KTHAD31B		15D	
16	15:35	44.01	3484	3.30			KTHAD31B		16D	
17	15:38	44.90	3466	3.22			KTHAD31B		17D	
18	15:41	46.90	3467	3.14			KTHAD31B		18D	
19	15:43	43.29	3486	3.20			KTHAD31B		19D	
20	15:46	49.07	3489	3.16			KTHAD31B		20D	
21	15:48	44.01	3502	3.12			KTHAD31B		21D	

22	15:51					KTHAD31B		22D	NO DATA
23	15:53	47.26	3454	3.15		KTHAD31B		23D	
24	15:56	44.90	3483	3.18		KTHAD31B		24D	
25	15:58					KTHAD31B		25D	
			Average	3.25					
			STDDEV	0.14					

AMMUNITION LOT NUMBER: KTHAD42A Dry   OAT 95 deg F

Propellant: WC 868

Projectiles: PGU-27/B

Loaded By: L. Wheeler

Barrel Serial Number: 026

Breech Block Serial Number: 008

KTHAD56	Al/MoO$_3$ Molar Ratio	Wt% Al/MoO$_3$	Wt% AN	Wt% Kel-F	Wt% C				OLD DIE SET Load Varied between 120 to 160 mg		
	2.63	66%	30%	2%	2%						
55	12:47	46.27	3382	3.51	580.0		KTHAD56	AMBIENT	Round 50		185mg
53	12:42	49.05	3390	9.45	580.0		KTHAD56	AMBIENT	Round 48		180mg
54	12:45	49.07	3394	3.50	580.0		KTHAD56	AMBIENT	Round 49		175mg
52	12:40	47.91	3388	3.57	580.0		KTHAD56	AMBIENT	Round 47		170mg
51	12:37	48.71	2752?	4.12	580.0		KTHAD56	AMBIENT	Round 46 Anomaly?		165mg
			Average	4.83	2.60						
46	12:24	47.3	3379	3.37	580.0		KTHAD56	AMBIENT	Round 41		160mg
47	12:26	49.43	3390	3.48	580.0		KTHAD56	AMBIENT	Round 42		
48	12:29	47.15	3380	3.21	580.0		KTHAD56	AMBIENT	Round 43		
49	12:31	48.35	2750?	3.55	580.0		KTHAD56	AMBIENT	Round 44 Instrument Anomaly		
50	12:34	46.54	3354	4.05	580.0		KTHAD56	AMBIENT	Round 45		
			Average	3.53	0.32						
41	10:48	47.91	3377	3.98	580.0		KTHAD56	AMBIENT	Round 36		155mg
42	10:51	49.43	3377	3.46	580.0		KTHAD56	AMBIENT	Round 37		
43	12:13	49.43	4567?	4.07	580.0		KTHAD56	AMBIENT	Round 38 Apparent Instrument Anomaly		
44	12:19	47.91	3390	3.34	580.0		KTHAD56	AMBIENT	Round 39		
45	12:22	49.43	3385	4.05	580.0		KTHAD56	AMBIENT	Round 40		
			Average	3.78	0.35						
36	10:36	47.98	3378	3.47	580.0		KTHAD56	AMBIENT	Round 31		150mg
37	10:38	48.67	3379	4.52	580.0		KTHAD56	AMBIENT	Round 32		
38	10:41	52.68	3392	3.76	580.0		KTHAD56	AMBIENT	Round 33		
39	10:43	51.06	3372	3.98	580.0		KTHAD56	AMBIENT	Round 34		
40	10:46	49.70	3379	4.08	580.0		KTHAD56	AMBIENT	Round 35		
			Average	3.96	0.39						

31	14:28	44.11	3355	3.63	580.0		KTHAD56	AMBIENT	Round 26 30 Sept 09	Old	145mg
32	14:32	46.18	3390	3.53	580.0		KTHAD56	AMBIENT	Round 27 30 Sept 09		
33	10:29	50.87	3400	3.39	580.0		KTHAD56	AMBIENT	Round 28 05 Oct 09		
34	10:31	49.07	3384	**5.80**	580.0		KTHAD56	AMBIENT	Round 29		
35	10:34	47.97	3377	3.36	580.0		KTHAD56	AMBIENT	Round 30		
			Average	3.94	1.04						

KTHAD54	Al/MoO$_3$ Molar Ratio	Wt% Al/MoO$_3$	Wt% AN	Wt% Kel-F	Wt% C			NEW AND OLD DIE SETS Load Varied between 120 to 160 mg
	2.63	66%	30%	2%	2%			

Date: 30 Sept 09 / 05 Oct 09   Test Engineer: D. Herigstad   Instrumentation: E. Gogley / L. Wheeler

Test: Green Primer Trials   Firing Officer: C. Lancaster   Gunner: A. Camacho / L. Wheeler (IT)

Event No.	Time	Peak Pressure (kpsi)	Velocity (fps)	Action Time (ms)	Prop. Wt. (grs)	Proj. Wt. (grs)	Primer Type	Barrel Temp (deg F)	Notes
1	13:29	44.87	3369	2.70	580.0		M52A3B1	AMBIENT	Checkout 1
2	13:38	61.22	3345	3.76	580.0		KTHAD54	AMBIENT	Round 1 30 Sept 09
3	13:48				580.0		M52A3B1	AMBIENT	Checkout 2 no data/ inst. No bueno
4	14:08	45.63	3369	2.75	580.0		M52A3B1	AMBIENT	Checkout 3
5	14:19	38.95	3347	2.75	580.0		M52A3B1	AMBIENT	Checkout 4
6	14:23	44.73	3371	3.83	580.0		KTHAD54	AMBIENT	Round 2 30 Sept 09
7	9:04	40.80	3363	2.77	580.0		M52A3B1	AMBIENT	Checkout 5 05 Oct 09
8	9:07	48.67	3392	5.03	580.0	160	KTHAD54	AMBIENT	Round 3
9	9:09	51.24	3411	3.74	580.0		KTHAD54	AMBIENT	Round 4
10	9:14	48.71	3383	23.28	580.0		KTHAD54	AMBIENT	Round 5
11	9:16	46.90	3384	3.35	580.0		KTHAD54	AMBIENT	Round 6
12	9:19	49.81	3404	3.88	580.0		KTHAD54	AMBIENT	Round 7
13	9:21	48.35	3391	3.44	580.0		KTHAD54	AMBIENT	Round 8
14	9:23	46.90	3378	3.73	580.0		KTHAD54	AMBIENT	Round 9
15	9:29	46.54	3376	4.06	580.0	155	KTHAD54	AMBIENT	Round 10
16	9:32	44.73	3370	3.65	580.0		KTHAD54	AMBIENT	Round 11
17	9:34	46.27	3364	3.73	580.0		KTHAD54	AMBIENT	Round 12
18	9:36	46.54	3391	3.74	580.0		KTHAD54	AMBIENT	Round 13
19	9:39	46.01	3372	3.84	580.0	150	KTHAD54	AMBIENT	Round 14
20	9:41	49.43	3390	14.64	580.0		KTHAD54	AMBIENT	Round 15
21	9:44	46.96	3378	4.23	580.0		KTHAD54	AMBIENT	Round 16
22	9:46	45.82	3379	3.77	580.0		KTHAD54	AMBIENT	Round 17
23	9:48	44.41	3367	3.5	580.0		KTHAD54	AMBIENT	Round 18
24	9:51	48.71	3365	12.87	580.0	145	KTHAD54	AMBIENT	Round 19

25	10:05	48.67	3365	9.63	580.0		KTHAD54	AMBIENT	Round 20
26	10:08	48.71	3398	4.15	580.0		KTHAD54	AMBIENT	Round 21
27	10:10	49.05	3402	4.78	580.0		KTHAD54	AMBIENT	Round 22
28	10:13	48.33	3384	5.1	580.0		KTHAD54	AMBIENT	Round 23
29	10:15	44.9	3376	4.17	580.0		KTHAD54	AMBIENT	Round 24
30	10:18	47.15	3392	4.05	580.0		KTHAD54	AMBIENT	Round 25
			STDDEV	6.16					

AMMUNITION LOT NUMBER: KTHAD 54 Dry   OAT 70

Propellant: WC 868

Projectiles: PGU-27A/B

Loaded By: A. Camacho / L. Wheeler

Barrel Serial Number: 004

Breech Block Serial Number: 008

KTHAD42A	Al/MoO$_3$ Molar Ratio	Wt% Al/MoO$_3$	Wt% AN	Wt% Kel-F	Wt% C				NEW DIE SET	
	2.69	64%	32%	2%	2%					

B52 BALLISTIC TEST LABORATORY
Code 478200D

										Instrumentation: L. Wheeler
Date: 28 August 09						Test Engineer: D. Herigstad				
Test: Green Primer Trials						Firing Officer: C. Lancaster				Gunner: A. Camacho
Event No.	Time	Peak Pressure (kpsi)	Velocity (fps)	Action Time (ms)	Prop. Wt. (grs)	Proj. Wt. (grs)	Primer Type	Barrel Temp (deg F)	Round Number	Notes
1	10:19	0	0	0	580.0		M52A3B1	Ambient	Chk Out 1	Instrumentation Error
2	10:49	47.62	0	2.73	580.0		M52A3B1	Ambient	Chk Out 2	V-Screen error
3	11:02	47.53	3419	2.78	580.0		M52A3B1	Ambient	Chk Out 3	
4	11:07	55.21	3423	28.01	579.4		KTHAD42A	Ambient	Round 6	
5	11:10	53.99	4010	4.71	580.2		KTHAD42A	Ambient	Round 7	
6	11:13	51.60	3949	43.04	579.9		KTHAD42A	Ambient	Round 26	
7	11:16	52.85	3388	33.81	580.6		KTHAD42A	Ambient	Round 27	
8	13:04	48.61	3399	4.72	579.1		KTHAD42A	Ambient	Round 1	
9	13:06	50.87	3403	18.03	580.2		KTHAD42A	Ambient	Round 2	
10	13:09	46.90	3393	5.1	580.5		KTHAD42A	Ambient	Round 3	
11	13:12	47.93	3388	4.6	580.9		KTHAD42A	Ambient	Round 4	
12	13:15	49.70	3443	4.09	580.1		KTHAD42A	Ambient	Round 5	
13	13:17	53.23	3404	24.68	580.41		KTHAD42A	Ambient	Round 8	
14	13:20	50.15	3389	25.71	580.26		KTHAD42A	Ambient	Round 9	
15	13:22	49.90	3389	7.18	580.86		KTHAD42A	Ambient	Round 10	
16	13:25	49.05	3395	8.98	580.04		KTHAD42A	Ambient	Round 11	
17	13:26	50.72	3365	38.05	579.12		KTHAD42A	Ambient	Round 12	
18	13:31	51.6	3391	21.45	580.86		KTHAD42A	Ambient	Round 13	
19	13:53	49.9	3395	12.76	580.32		KTHAD42A	Ambient	Round 14	
20	13:56	49.81	3383	31.69	580.12		KTHAD42A	Ambient	Round 15	

21	13:58	51.33	3405	5.32	579.99		KTHAD42A	Ambient	Round 28	
22	14:01	51.33	3383	26.67	580.12		KTHAD42A	Ambient	Round 29	
23	14:03	50.57	3380	14.76	580.61		KTHAD42A	Ambient	Round 30	
			STDDEV	15.69						
AMMUNITION LOT NUMBER: KTHAD42A Dry					OAT 102 deg F					
Propellant: WC 868										
Projectiles: PGU-27/B										
Loaded By: A. Camacho / L. Wheeler										
Barrel Serial Number: 004										
Breech Block Serial Number: 008										